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AMERICAN SOCIETY
for
TESTING MATERIALS

PROCEEDINGS
of the
FORTY-FIFTH ANNUAL MEETING

Held at Atlantic City, N. J.

June 22–26, 1942

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VOLUME 42

COMMITTEE REPORTS

TECHNICAL PAPERS

Published by the
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SUMMARY OF PROCEEDINGS OF THE FORTY-FIFTH ANNUAL MEETING

ATLANTIC CITY, N. J., JUNE 22–26, 1942

The following Summary records the action taken by the Society at the Annual Meeting on the Recommendations Affecting Standards and Tentative Standards appearing in the annual reports of the standing committees. In the individual committee reports attention is called through an editorial footnote to any modifications of the recommendations or where the recommendations of the committee were not approved. In all other instances it is to be understood that the recommendations were approved as submitted by the committee. The new and revised standards and tentative standards are published in the 1942 Book of A.S.T.M. Standards, Parts I, II, and III. Except as noted, the reports and papers appear in this volume.

This summary of the Forty-fifth Annual Meeting of the American Society for Testing Materials, held at Chalfonte—Haddon Hall, Atlantic City, N. J., June 22–26, 1942, is a record of the transactions of the meeting including the actions taken on the various recommendations submitted by the standing committees. In all, 16 sessions were held.

The Proceedings are set forth session by session. There were 60 reports and 56 formal papers presented. The record with respect to each has been briefed, the recommendations in the reports having been grouped so as to cover the acceptance of material for publication as tentative, such as new specifications, methods of test, revisions of tentative standards and proposed revisions of existing standards, and as a separate group, the approval of matters that were referred to letter ballot of the Society, comprising the adoption of tentative standards as standard and the adoption of revisions of standards. Accordingly, wherever the action is reported as "adopted as standard," it is understood that this indicates approval of the annual meeting for reference to letter ballot of the Society. The various recommendations so recorded are included in the Society letter ballot.[1] The actions designated as "accepted as tentative" are self evident as indicating acceptance by the Society at the annual meeting for publication as tentative.

While all of the items on the program are recorded under the particular session in which they were presented, for convenience in locating actions with respect to any particular report, the accompanying list is presented of all the reports together with the page reference where the action thereon is recorded.

The registered attendance of the meeting is as follows: Members present or represented, 835; committee members, 264; guests, 275; total, 1374; ladies, 219.

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[1] The letter ballot on recommendations affecting standards, distributed to the Society membership, was canvassed on September 1, 1942 and all actions were approved.

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FIRST, SECOND, AND FIFTH SESSIONS—SYMPOSIUM ON RADIOGRAPHY

TUESDAY, JUNE 23, 9.30 A.M., 1 P.M., AND 8 P.M., RESPECTIVELY

President G. E. F. Lundell, *chairman*.

President Lundell, in opening the Forty-fifth Annual Meeting extended a very cordial welcome to all members and guests of the Society in attendance. He expressed the belief that from attendance records and other indications, this meeting promised to be one of the most successful in the history of the Society, and that this was most encouraging considering that the membership as a whole has so many activities at the present time. President Lundell referred briefly to the complete program for the meeting replete with symposiums, round table discussions and technical sessions at which the technical papers and annual reports of the committees would be presented, many of them having a direct bearing on the war effort. He also stated that the scheduled meetings of standing committees and their subcommittees gave promise of further valuable contributions by the Society in the standardization and promotion of knowledge of engineering materials.

Committee E-7 on Radiographic Testing:
A brief oral progress report was presented by H. H. Lester, chairman.

Symposium on Radiography:
President Lundell in opening the Symposium on Radiography, the second held at an annual meeting of the Society, referred to the timely nature of such an interesting series of papers which he indicated should help to clarify the procedures and applications of this important method of testing, particularly in the testing and inspection of war materials. This symposium was arranged in order to acquaint the engineering profession with the most recent developments in X-ray and radium radiography of engineering materials and to provide those interested with an opportunity to discuss and coordinate their ideas and methods of procedure.

Since radiography as a tool for testing materials is relatively new, numerous problems have arisen since the first Symposium was held in 1936,[2] partly brought about by the phenomenal increase in the applied voltage and a more extended use of radium. There has also been a diversification with regard to the products now studied by radiography due to the increased war industry which has given rise to numerous problems. Some radiographers have been more successful in solving these problems than others, and an exchange of ideas provided by such a symposium might contribute considerably to the war effort. The papers comprising the symposium were then presented at the three sessions, as follows:

First Session Co-Chairmen: Lars Thomassen; Gilbert E. Doan

Some Aspects of X-ray Inspection Applied to Testing and Production—Don M. McCutcheon, presented from manuscript by the author.

Radiography of Welds and Weldments—R. E. Lorentz, Jr., presented from manuscript by the author.

Some Calibration Data and Scatter Measurements for the Radiography of Magnesium Aircraft Castings—L. W. Ball, presented from manuscript by the author.

A Correlation of the Mechanical Properties and Radiographic Appearance of Magnesium Alloy Castings—R. S. Busk, presented by the author.

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[2] Symposium on Radiography and X-ray Diffraction Methods, Thirty-ninth Annual Meeting, June 30–July 1 1936. (Symposium issued as separate publication.)

Second Session Co-Chairmen: Herman E. Seemann; James J. Curran.

Application of High Voltage X-rays in the Boiler Shop—O. R. Carpenter, presented from manuscript by the author.

The Gamma-Ray Radiography of Welded High-Pressure Power Plant Piping—R. W. Emerson, presented by the author.

Contrast and Latitude in Steel Casting Radiography with Million-Volt X-rays—C. D. Moriarty, presented in abstract by W. A. Williams.

Methods of Evaluating the Qualities of the New Type X-ray Films and Equipment—T. A. Triplett and Vance Danford, presented in abstract by Mr. Danford.

An Investigation of the Apparatus Used in Radium Radiography—L. W. Ball and D. R. Draper, presented from manuscript by Mr. Ball.

Fifth Session Co-Chairmen: J. T. Norton; R. A. Gezelius.

An Exposure Meter for X-ray Radiography—Herbert Friedman and Arthur L. Christenson, presented from manuscript by Mr. Friedman.

A Study of Cassette Design for the Radiography of Aircraft Castings—L. W. Ball, presented from manuscript by the author.

Equivalent Penetrameters in Radiographic Testing—Robert J. Schier and Gilbert E. Doan, presented from manuscript by Mr. Doan.

Precision Radiography—III—Robert J. Schier and Gilbert E. Doan, presented from manuscript by Mr. Doan.

A Million-Volt Portable X-ray Unit—Ernest E. Charlton and W. F. Westendorp, presented from manuscript by Mr. Westendorp.

THIRD SESSION—ELECTRICAL INSULATING MATERIALS, PAINT, RUBBER, TEXTILES

TUESDAY, JUNE 23, 2 P.M.

Session Co-Chairmen: M. F. Skinker; Vice-President H. J. Ball.

Sectional Committee C59 on Electrical Insulating Materials:

Report presented by H. L. Curtis, chairman.

Committee D-9 on Electrical Insulating Materials:

Report presented by T. Smith Taylor, chairman, and the following actions taken:

Accepted as Tentative:

Test for Sludge Formation in Mineral Transformer Oil
Tests for Steatite Used as Electrical Insulation
Tests for Vulcanized Fibre Used for Electrical Insulation (to replace Tentative Methods D 619 - 41 T)
Methods of Measuring Dimensions of Rigid Tubes Used in Electrical Insulation
Test for Power Factor and Dielectric Constant Parallel with Laminations of Laminated Sheet and Plate Insulating Materials

Revisions of:

Spec. for Molds for Test Specimens of Molding Materials Used for Electrical Insulation (D 647 - 41 T)
Test for Compressive Strength of Electrical Insulating Materials (D 649 - 41 T)
Test for Flexural Strength of Electrical Insulating Materials (D 650 - 41 T)
Test for Tensile Strength of Electrical Insulating Materials (D 651 - 41 T)
Tests for Electrical Porcelain (D 116 - 39)
Tests for Sheet and Plate Materials Used in Electrical Insulation (D 229 - 39)
Tests for Laminated Tubes Used in Electrical Insulation (D 348 - 39)
Tests for Laminated Round Rods Used in Electrical Insulation (D 349 - 39)
Tests for Shellac Used for Electrical Insulation (D 411 - 40)

Adopted as Standard:

Test for Thickness of Solid Electrical Insulation (D 374 - 41 T)
Tests for Pin-Type Lime Glass Insulators (D 468 - 39 T), as revised
Test for Water Absorption of Plastics (D 570 - 40 T), approved jointly with Committee D-20 on Plastics

Revisions of:

Test for Arc Resistance of Solid Electrical Insulating Materials (D 495 - 41)
Tests for Electrical Insulating Oils (D 117 - 40), the tentative revision which appeared in the report as preprinted, was recommended for immediate adoption as standard and was accepted by unanimous consent, subject to favorable letter ballot of Committee D-9,[3] with the following additional change:

Section 15.—Add the following as a new Section 15, renumbering the remaining sections accordingly:

> 15. *Saponification Number.*—Saponification number shall be determined in accordance with the Tentative Method of Test for Saponification Number (D 94 - 41 T) of the American Society for Testing Materials.

Tests for Electrical Porcelain (D 116 - 39)
Tests for Shellac Used for Electrical Insulation (D 411 - 40)
Tests for Sheet and Plate Materials Used in Electrical Insulation (D 229 - 39)
Tests for Laminated Tubes Used in Electrical Insulation (D 348 - 39)
Tests for Laminated Round Rods Used in Electrical Insulation (D 349 - 39)

Withdrawal of Standards:

Tests for Molded Materials Used for Electrical Insulation (D 48 - 39)
Test for Impact Resistance of Electrical Insulating Materials (D 256 - 38)

Editorial Change Accepted:

Tests for Sheet and Plate Materials Used in Electrical Insulation (D 229 - 39)

The paper by A. E. Flowers and S. A. Fruchtman, on "A Statistical Comparison of the Methods for the Determination of the Oxidation Tendency of Insulating Oils," to be appended to the report, was presented from manuscript by Mr. Flowers.

Committee D-1 on Paint, Varnish, Lacquer, and Related Products:

Report presented by H. E. Smith chairman, and the following action taken:

Accepted as Tentative:

Spec. for C. P. Toluidine Toner
Spec. for Chrome Yellow and Chrome Orange, to replace Standard D 211 - 41
Test for Abrasion Resistance of Organic Coatings with the Air Blast Tester, with an editorial change to include an illustration of the glass nozzle
Test for Spectral Characteristics and Color of Objects and Materials, with editorial changes, to replace Standard D 307 - 39
Test of Consistency of Exterior House Paints and Enamel Type Paints, to replace Standard D 562 - 41

Revisions of Methods of Sampling and Testing Aluminum Powder and Paste (D 480 - 41 T)
Revisions of Def. of Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16 - 41 T)

Adopted as Standard:

Spec. for Barium Sulfate Pigments (D 602 - 41 T)
Spec. for Mica Pigment (D 607 - 41 T)
Spec. for Diatomaceous Silica Pigment (D 604 - 41 T)
Spec. for Aluminum Silicate Pigment (D 603 - 41 T)
Spec. for Lead Titanate (D 606 - 41 T)
Spec. for Magnesium Silicate Pigment (D 605 - 41 T), as revised
Revision of Methods of Sampling and Testing Lacquer Solvents and Diluents (D 268 - 41)

Withdrawal of Standards:

Spec. for Chrome Yellow (D 211 - 41)
Test for Spectral Apparent Reflectivity of Paints (D 307 - 39)
Test for Consistency of Enamel Type Paints (D 562 - 41)

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[3] Results of the letter ballot on this recommendation, indicating its approval, are given in the report of Committee D-9. See p. 366.

Committee D-11 on Rubber Products:

Report presented by O. M. Hayden, chairman, and the following actions taken:

Accepted as Tentative:

Test for Indentation of Rubber by Means of the Durometer

Adopted as Standard:

Test for Viscosity and Total Solids Content of Rubber Cements (D 553 - 39 T)
Revisions of Test for Accelerated Aging of Vulcanized Rubber by the Oxygen-Pressure Method (D 572 - 41)
Revisions of Test for Accelerated Aging of Vulcanized Rubber by the Oven Method (D 573 - 41)

The report on "Digest of Cooperative Test Program on Accelerated Light Aging of Rubber," prepared by J. H. Ingmanson, appended to the report, was presented by title.

Committee D-12 on Soaps and Other Detergents:

Report presented by H. P. Trevithick, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Compound Chip Soap (with Rosin)
Spec. for Compound Powdered Soap (Granulated, with Rosin)
Spec. for Built Soap, Powdered (Type D)
Revisions of Spec. for Tetrasodium Pyrophosphate (Anhydrous) (D 595 - 40 T)
Revisions of Def. of Terms Relating to Soaps and Other Detergents (D 459 - 41 T)
Revisions of Methods of Sampling and Chemical Analysis of Soaps and Soap Products (D 460 - 41)

Adopted as Standard:

Spec. for Olive Oil Solid Soap (Type A, Pure; Type B, Blended) (D 592 - 41 T)
Spec. for Olive Oil Chip Soap (Type A, Pure; Type B, Blended) (D 630 - 41 T)
Spec. for Palm Oil Chip Soap (Type A, Pure; Type B, Blended) (D 536 - 41 T)
Spec. for Salt-Water Soap (D 593 - 40 T)
Test for Carbonates as Carbon Dioxide in Soaps and Soap Products (Evolution-Volumetric Method) (D 460 - 41 T)
Revisions of Spec. for Alkaline Soap Powder (D 534 - 41)
Revisions of Methods of Sampling and Chemical Analysis of Soaps and Soap Products (D 460 - 41)

Committee D-13 on Textile Materials:

Report presented, by W. H. Whitcomb, secretary, and the following actions taken:

Accepted as Tentative:

Spec. for Woven Asbestos Cloth
Spec. for Finished All-Cotton Upholstery Tapestries
Tests and Tolerances for Certain All-Cotton and Cotton-and-Rayon Fine Fancy Goods
Tests and Tolerances for Certain Fine Staple Gray Goods
Tests and Tolerances for Jute Rove and Plied Yarn for Electrical and Packing Purposes
Test for Colorfastness to Atmospheric Gases of Dyed Cellulose Acetate Rayon
Test for Identification of Finishes on Textiles
Resistance of Textile Fabrics to Microorganisms
Revisions of:
Tests and Tolerances for Woven Tapes (D 259 - 39 T)
Tests and Tolerances for Certain Wool and Part Wool Fabrics (D 462 - 37 T)
Methods of Quantitative Analysis of Textiles (D 629 - 41 T)
Tests for Wool Felt (D 461 - 40)
Spec. and Test for Fineness of Wool Tops (D 472 - 41)

Adopted as Standard:

Test for Commercial Weight of Continuous Filament Rayon Yarns (D 258 - 41 T), as revised
Test for Resistance to Yarn Slippage in Silk, Rayon, and Silk-Rayon Woven Fabrics (D 434 - 36 T)
Tests for Rayon Staple (D 540 - 41 T)
Test for Commercial Weight of Spun Rayon Yarns and Threads (D 507 - 41 T), as revised
Spec. and Test for Asbestos Yarns (D 299 - 40 T)
Testing Woven Asbestos Cloth (D 577 - 40 T)
Revisions of:
Tests and Tolerances for Hose Ducks and Belt Ducks (D 181 - 40)
Tests and Tolerances for Continuous Filament Rayon Yarns (D 258 - 40)

Adopted as Standard:

Revisions of:

Tests for Pile Floor Covering (D 418 – 40)
Spec. and Test for Asbestos Roving for Electrical Purposes (D 375 – 41)
Test for Fastness of Dyed or Printed Cotton Textiles to Laundering or Domestic Washing (D 435 – 41), in the form of a new Standard Method of Test for Colorfastness to Commercial Laundering and to Domestic Washing of Cotton and Linen Textiles
Testing and Tolerances for Cotton Yarns (D 180 – 41)
Testing and Tolerances for Cotton Sewing Threads (D 204 – 39)
Testing and Tolerances for Tire Cord, Woven and on Cones (D 179 – 38)
Spec. for Textile Testing Machines (D 76 – 41)
Def. of Terms Relating to Textile Materials (D 123 – 41)

Withdrawal of Standard:

Test for Estimating Hard Scoured Wool in Wool in the Grease (Mill Scale Operations) (D 232 – 36)

Reaffirmation of Standards:

Spec. for Tolerances for Numbered Cotton Duck (D 230 – 27)
Spec. for 0.007-in. Cotton Tape for Electrical Purposes (D 335 – 36)
Spec. and Test for Holland Cloth (D 376 – 35)
Tests and Tolerances for Certain Light and Medium Weight Cotton Fabrics (D 274 – 36)
Method of Determining Relative Humidity (D 337 – 34)
Test for Shrinkage in Laundering of Woven Cotton Cloth (D 437 – 36)

Sectional Committee L14 on Fastness of Colored Textiles:

Report presented, in the absence of the chairman, by the chair.

Papers:

The following papers were presented:

A New Recording Viscometer for Paint Consistency Measurements[4]—C. R. Wicker and J. A. Geddes, presented by D. H. Dawson.
An Analysis of the Uranyl Oxalate Actinometer as Applied to Accelerated Light and Weathering Tests—F. T. Bowditch, C. E. Greider and C. G. Ollinger, presented by Mr. Ollinger.

FOURTH SESSION—WATER

TUESDAY, JUNE 23, 2 P.M.

Session Co-Chairmen: President G. E. F. Lundell, Max Hecht

Committee D-19 on Water for Industrial Uses:

Report presented by F. N. Speller, vice-chairman, and the following actions taken:

Adopted as Standard:

Test for Determination of the Calcium Ion and Magnesium Ion in Industrial Waters (D 511 – 40 T)
Test for Determination of the Chloride Ion in Industrial Waters (D 512 – 40 T)
Test for Determination of the Sulfate Ion in Industrial Waters (D 516 – 40 T)

Mr. Speller then called upon D. K. French to describe the activities of Subcommittee III on Methods of Sampling, on H. C. Miller to report on the work of Subcommittee IV on Methods of Analysis, and on W. C. Schroeder to report on the work of Subcommittee V on Classification.

Report of Joint Research Committee on Boiler Feedwater Studies: Report not preprinted presented from manuscript by C. H. Fellows, chairman.

Round-Table Discussion on the Solvent Action of Water Vapor at High Temperature and Pressure:

This Round-Table Discussion was sponsored by Committee D-19 on Water for Industrial Uses as a further contribution in its program of presenting timely reviews in its special field of testing dealing with industrial waters. Following the Symposium on Problems and Practice in Determining Steam Purity by Conductivity Methods held last year the committee planned this informal round-table session in order to develop information on the quantitative determination of materials carried along in boiler water or steam that contribute to deposit formation. There are numerous questions as to the nature and source of these deposits such as: How is this material transferred from the boiler water to its final location in superheaters and turbines? Is mechanical carry-over the sole explanation? What is the mechanism of its deposition? Why is silica found soluble at times, combined as sodium silicate; at other times, insoluble, uncombined, as crystalline quartz here and amorphous silica-glass there? What may we find in the physical chemistry of high temperatures and pressures which may help to explain what we actually find? These and many other questions were considered at length by those who were invited to participate in the discussion.

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[4] Published in ASTM BULLETIN, No. 120, January 1943, p. 11.

Following the introduction to the Round-Table Discussion, presented by George W. Morey, Geophysical Laboratory, Carnegie Institution of Washington, prepared discussions were presented by the following, after which those attending the session participated:

L. Drew Betz, W. H. and L. D. Betz
B. O. Buckland, General Electric Co.
Joint Research Committee on Boiler Feedwater Studies, C. H. Fellows, The Detroit Edison Co.
R. E. Hall, Hall Laboratories, Inc.
J. A. Holmes, National Aluminate Corp.
F. R. Owens, Cyrus W. Rice and Co.
P. B. Place, Combustion Engineering Co. Inc.
S. T. Powell, Consulting Chemical Engineer
A. H. Reynolds, Dearborn Laboratories
J. B. Romer, Babcock & Wilcox Co.
F. G. Straub, University of Illinois
S. E. Tray, Allis Chalmers Manufacturing Co.

FIFTH SESSION—SYMPOSIUM ON RADIOGRAPHY

(*Continued from Second Session*)

TUESDAY, JUNE 23, 8 P.M.

This session was a continuation of the Symposium on Radiography. The papers presented in this session are listed previously with those in the First and Second Sessions.

SIXTH SESSION—PLASTICS, TIMBER

TUESDAY, JUNE 23, 8 P.M.

Session Chairman: W. E. Emley.

Committee D-20 on Plastics:

Report presented by Bjorn Andersen, first vice-chairman, and the following actions taken:

Accepted as Tentative:

Recommended Practice for Repeated Flexural Stress (Fatigue) of Plastics, with several editorial modifications, including a change in the title to read "Tentative Method of Test" rather than "Tentative Recommended Practice."

Accepted as Tentative:

Test for Mar Resistance of Plastics
Test for Measuring Photoelectrically the Haze of Transparent Plastics
Rec. Practice for Long-Time Tension Tests of Plastics
Descriptive Nomenclature for Objects Made from Plastics

Adopted as Standard:

Test for Index of Refraction of Transparent Organic Plastics (D 542 - 40 T)
Test for Water Absorption of Plastics (D 570 - 40 T), jointly with Committee D-9 on Electrical Insulating Materials

Committee D-6 on Paper and Paper Products:

Report presented, in the absence of the chairman, by G. H. Harnden, secretary, and the following actions taken:

Accepted as Tentative:

Test for Pentosans in Paper
Test for Conditioning Paper and Paper Products for Testing
Test for Quantitative Determination of Coating on Mineral-Coated Paper
Test for Internal Tearing Resistance of Paper, with an editorial change in the first sentence and formula in Section 4 (*c*) providing for the interchanging of the symbols *H* and *h*.
Test for Qualitative Examination of Mineral Fillers and Mineral Coatings of Paper
Revisions of Test for Basis Weight of Paper and Paper Products (D 646 - 41 T)
Revisions of Test for Moisture in Paper (D 644 - 41 T)

Adopted as Standard:

Test for Sampling of Paper and Paper Products (D 585 - 40 T)
Test for Ash Content of Paper and Paper Products (D 586 - 40 T)
Test for Casein in Paper (Qualitative) (D 587 - 40 T)
Test for Alpha-, Beta-, and Gamma-Cellulose in Paper (D 588 - 40 T)
Test for Paraffin Content of Waxed Paper (D 590 - 40 T)
Test for Starch in Paper (D 591 - 40 T)

Committee D-7 on Timber:

Report presented, in the absence of the chairman, by L. J. Markwardt, and the following action taken:

Adopted as Standard:

Revision of Test for Distillation of Creosote (D 246 - 39)

Committee C-5 on Fire Tests of Materials and Construction:

Progress report not preprinted presented from manuscript, in the absence of the chairman, by the chair.

Committee D-17 on Naval Stores:

Report presented, in the absence of the chairman, by V. E. Grotlisch, and the following actions taken:

Adopted as Standard:

Test for Saponification Number of Rosin (D 464 - 37 T)
Test for Acid Number of Rosin (D 465 - 37 T)

The recommendation, not preprinted, for adoption of the above two tentative standards was accepted by unanimous consent, subject to favorable letter ballot of Committee D-17,[5] with the inclusion in each method of the following note:

> Section 1.—Add the following as a note at the end of the section on Scope:
>
> NOTE.—Due to difficulty in observing the end point in the titration, because of the dark color of the solution, this method using an internal indicator is not recommended for use with rosins of grades darker than G, that is, of grades D, E, F, and FF.

Papers:

The following papers were presented:

Mar Resistance of Plastics—Ladislav Boor, presented by the author.
Mechanical Tests of Cellulose Acetate—Part II on Creep—William N. Findley, presented by the author.
Acid and Alkali Resistance of Plastics[6]—John Delmonte, presented from manuscript by the author.
The Influence of Temperature on the Mechanical Properties of Molded Phenolic Materials—T. S. Carswell, D. Telfair and R. U. Haslanger, presented by H. K. Nason.
Stress-Strain Relations in Timber Beams (Douglas Fir)—Albert G. H. Dietz,[6a] presented by title.
Method for Inspection and Control of Liquid Starch-Base Adhesives for Bottle Labeling and Case Sealing—A. Herman and F. M. Knowlton, presented by Mr. Knowlton.

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[5] Results of the letter ballot on this recommendation, indicating its approval, are given in the report of Committee D-17, see p. 442.

[6] Published in ASTM BULLETIN, No. 119, December, 1942, p. 11.

[6a] Published in ASTM BULLETIN, No. 118, October, 1942, p. 19.

SEVENTH SESSION—ROUND-TABLE DISCUSSION ON ALTERNATE ALLOY STEELS AND CONSERVATION OF STRATEGIC ALLOYS

WEDNESDAY, JUNE 24, 9.30 A.M.

Session Chairman: John Mitchell.

In order to provide the latest information with respect to the vital subject of alternate alloy steels, this round-table discussion was arranged, in which important developments were presented by eight discussion leaders as indicated below. Opportunity was also given for discussion or questions from those attending the session.

H. J. French, War Production Board—The status of raw materials considered necessary for steel manufacture.

C. H. Herty, Jr., Bethlehem Steel Co.—What has been accomplished by the steel manufacturers to conserve manganese and aluminum or other strategic elements considered necessary for steel manufacture.

N. I. Stotz, Universal-Cyclops Steel Corp.—Steps taken by the tool steel manufacturers to conserve elements considered necessary for tool steels.

V. W. Whitmer, Republic Steel Corp.—The application of stainless steels to conserve those elements considered necessary for corrosion of heat-resisting alloys.

C. M. Parker, American Iron and Steel Institute—The cooperative work of the A. I. S. I. and other technical societies; how the National Emergency Steels were established, and what measures had to be considered in establishing National Emergency Steels to effect conservation.

W. P. Eddy, Yellow Truck and Coach Manufacturing Co.—The War Production Board Committees, how they were set up, what duties they were expected to perform, what progress has been made, what remains to be done, and other general details concerning the Alloy Bar, Billet and Bloom Committee now in operation.

A. W. F. Green, Pratt & Whitney Aircraft Division, United Aircraft Corp.—What has been accomplished in the aircraft engine industry to conserve strategic elements; what steps must be taken by engine producers to change parts; what testing is now in progress; what is being accomplished by the frame manufacturers to use sheet steel in place of aluminum.

M. J. R. Morris, Republic Steel Corp.—The application of National Emergency Steels and what the future holds for further work to effect the conservation.

While this round-table discussion was originally scheduled as an informal or off-the-record presentation, it has been possible, with the approval of the chairman, Mr. John Mitchell, and the discussion leaders to publish the contributions of all but two of those who participated in the formal part of the program. These six papers appear in the ASTM BULLETIN, No. 117, August, 1942, pages 29 to 43.

EIGHTH SESSION—FUELS AND LUBRICANTS; ROAD AND PAVING MATERIALS; BITUMINOUS MATERIALS

WEDNESDAY, JUNE 24, 1.30 P.M.

Session Chairman: H. S. Mattimore.

Committee D-5 on Coal and Coke:

Report presented, in the absence of the chairman, by W. A. Selvig, secretary, and the following actions taken:

Adopted as Standard:

Revision of Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 - 40) pertaining to tolerances for volatile matter determination in Section 43.

Committee D-3 on Gaseous Fuels:

Report presented by A. W. Gauger, chairman.

Committee D-2 on Petroleum Products and Lubricants:

Report presented by T. A. Boyd,

chairman, and the following actions taken:

Accepted as Tentative:

Test for Neutralization Number of Petroleum Products by Color-Indicator Titration

Test for Neutralization Number of Petroleum Products by Electrometric Titration

Test for Rust-Preventing Characteristics of Steam-Turbine Oils in Presence of Water

Method for Conversion of Kinematic Viscosity to Saybolt Furol Viscosity, this recommendation, not preprinted, was accepted by unanimous consent, subject to favorable letter ballot of Committee D-2.[7]

Revision of Test for Gum Stability of Gasoline (D 525 - 41 T), with the following additional change, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee D-2:[7]

Section 3 (b).—Change the last sentence from the form as preprinted to read as follows by the omission of the words and figures in brackets:

> If [the pressure in the bomb does not change more than 1 psi. in 10 ± 2 min., it may be assumed that] there are no leaks [and], the test may be continued.

Note 6.—In place of omitting this note, as recommended in the preprint, change it to read as follows:

> NOTE 6.—An initial rapid drop in pressure, generally not over 6 psi., may be observed because of the solution of oxygen in the sample. If after a few minutes the rate of pressure drop does not exceed 1 psi. in 10 min., the absence of leaks may be assumed.

Revisions of Test for Knock Characteristics of Motor Fuels (D 357 - 41 T)

Revisions of Test for Saponification Number (D 94 - 41 T)

Revisions of Test for Kinematic Viscosity (D 445 - 39 T)

Revisions of Test for Knock Characteristics of Aviation Fuels (D 614 - 41 T), with the following changes, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee D-2:[7]

Footnote 2.—Change the second sentence of the second paragraph to read as follows by the addition of the italicized words and the omission of those in brackets:

> Based on tests of a variety of aviation fuels in current aircraft engines and on service experience, this method [satisfactorily] rates such fuels [for purposes of procurement and use under normal operating conditions] *under conditions approximating best power, but is not intended to correlate with full-rich take-off conditions*.

Section 4 (b).—In the first sentence, delete the word "leaded" in the phrase "secondary reference fuels"; also delete the word "unleaded" in footnote 5.

Section 5 (c).—Delete this paragraph, relettering the subsequent paragraphs accordingly.

Section 5 (g).—Reletter as Paragraph (*h*) and add a new paragraph to read as follows:

> (*g*) *Adjustment of Micrometer*.—When the clearance volume to the top face of the therma-plug hole is 140 ± 0.5 ml., as measured by the "tilt" method (see Appendix), with the piston at top dead center, the micrometer shall be set to read 0.500 in.

Section 5 (i).—In the second sentence insert the reference "(Appendix)" after the words, "compression ratio." Add the following Note 1 at the end of the first paragraph with reference at the end of the first sentence:

> NOTE 1.—For routine purposes a blend of certified secondary reference fuels may be used instead of the blend of primary reference fuels.

Section 5 (k).—Change the requirement for breaker-point gap from "0.015 in." to read "0.020 in."

Section 5 (m).—Change from its present form to read as follows:

> *Valve Clearances*.—Hot and running clearances shall be 0.008-in. intake and 0.006-in. exhaust with the engine running under standard operating conditions on a reference fuel of 87 octane number.
>
> NOTE 2.—The valves may be adjusted with the engine stopped, either hot or cold, if appropriate allowances are made so that the hot and running clearances will be as specified.

Section 5 (q).—At the end add a reference "See Section A5 (*a*)," and a new paragraph reading as follows:

> When air of the above humidity prevails in the engine room through the operation of natural factors or the use of air conditioning, it may be used without further conditioning. Its humidity shall be determined by use of the type of sling psychrometer currently approved by the U. S. Weather Bureau.

Section 12.—Add a section on "Reproducibility of Results" identical with Section 14 of proposed revision of Method D 357 - 41 T.

Section A1.—Change the limitation on atmospheric pressures to read "not less than 26.0 in. (660 mm.) of mercury."

Section A2.—Insert the word "minimum" after "exhaust pipe."

Section A4 (e).—Add the following note after this section on valve timing:

> NOTE.—The recommended method consists of the following steps:
>
> 1. Remove the intake and exhaust push rods.
> 2. Swing the intake and exhaust rocker arms to a vertical position.
> 3. Mount a dial indicator on the rocker-arm support bracket with the indicating plunger directly over and pointing at the intake tappet.
> 4. Place a piece of drill rod between the intake tappet and the indicator plunger. This rod should be approximately ¼ in. in diameter, have a spherical end where it rests in the tappet, and have an indentation on the other end in which the indicating plunger will rest.

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[7] Results of the letter ballot on this recommendation indicating its approval, are given in the report of Committee D-2, see p. 307.

5. Set dial gage position so its plunger is approximately in the middle of its travel when the tappet is on the base circle of the cam. Then set the dial indicator at zero.

6. Rotate the flywheel in its running direction until the indicator shows that the cam is lifting the tappet. Mark the flywheel directly under the T.D.C. pointer at the angle where the tappet has been lifted 0.010 in. Repeat this operation two or three times to make sure of the angle at which this 0.010-in. lift is obtained. This is the point at which the effective valve opening starts. The part of the cam below the 0.010-in. lift is the quieting ramp and does not contribute to the effective valve opening.

7. This mark should be at the timing line on the flywheel marked "I. O. 0.010" which is 10 deg. a.t.d.c.

8. If the cam timing is not within ⅜ in. of the mark on the flywheel, the camshaft will need retiming either by shifting the cam gear with respect to the crankshaft gear or by relocating the cam gear on its shaft by using one of the other two keyways provided. Shifting the timing one full gear tooth will make 1.48-in. change on the flywheel. The extra keyways provided on the camshaft permit adjusting the timing in 0.37-in. and 0.74-in. increments.

Section A4 (f).—Change to read as follows:

(*f*) *Valve Clearances.*—Hot and running clearances shall be 0.008-in. intake and 0.006-in. exhaust with the engine running under standard operating conditions on a reference fuel of 87 octane number. To avoid possible change in clearances throughout the compression-ratio range, set the top linkage of the rocker-arm carrier and the rocker arms in a horizontal plane at 0.500 micrometer setting with valves closed.

Section A4 (i).—Insert a new paragraph reading as follows:

Micrometer.—A suitably mounted micrometer for measuring the height of the cylinder with respect to the crankcase.

Section A6 (c).—Change spark plug type to RJ-11-64-A.

Section A7.—Designate as Paragraph (*a*) and add a new Paragraph (*b*) reading as follows:

(*b*) Ethylene glycol shall be used as coolant. A maximum of 1 per cent by weight or by volume of a suitable corrosion inhibitor may be added to the ethylene glycol.[a]

Section A13 (e).—Delete the second sentence.

Appendix.—Insert a new Appendix identical with that being added in the proposed revision of Method D 357 - 41 T.

Adopted as Standard:

Test for Carbon Residue of Petroleum Products (Ramsbottom Carbon Residue) (D 524 - 41 T)

Test for Tetraethyl Lead in Gasoline (D 526 - 41 T)

Test for Dropping Point of Lubricating Grease (D 566 - 40 T), this recommendation, not preprinted, was accepted by unanimous consent, subject to favorable letter ballot of Committee D-2.[7]

Revisions of Test for Gum Content of Gasoline (D 381 - 36)

Revisions of Test for Flash Point by Means of the Pensky-Martens Closed Tester (D 93 - 40)

Revisions of Test for Melting Point of Paraffin Wax (D 87 - 37)

Revisions of Test for Vapor Pressure of Petroleum Products (Reid Method) (D 323 - 41), with the following additional changes, to clarify the description of the apparatus, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee D-2:[7]

Section 2 (a), (b) and (c).—Revise to read as follows:

2. (*a*) *Air Chamber.*—The upper section or air chamber, shown in Fig. 1(*c*) shall be a cylindrical vessel $2\frac{1}{16} \pm \frac{1}{16}$ in. in diameter and $10 \pm \frac{1}{8}$ in. in length, inside dimensions, with the inner surfaces of the ends slightly sloped to provide complete drainage from either end when held in a vertical position. On one end of the air chamber, a suitable gage coupling with internal diameter not less than $\frac{3}{16}$ in. shall be provided to receive the ¼-in. gage connection. In the other end of the air chamber an opening approximately ½ in. in diameter shall be provided for coupling with the gasoline chamber. Care shall be taken that the connections to the end openings do not prevent the chamber from draining completely.

(*b*) *Gasoline Chamber.*—The lower section, or gasoline chamber, shown in Fig. 1(*a*), shall be a cylindrical vessel of the same inside diameter as the air chamber and of such volume as to fulfill the requirements of Paragraph (*c*). In one end of the gasoline chamber an opening approximately ½ in. in diameter shall be provided for coupling with the air chamber. The inner surface of the end containing the coupling member shall be sloped to provide complete drainage when inverted. The other end of the gasoline chamber shall be completely closed.

(*c*) *Gasoline Chamber for Sampling from Closed Vessels.*—For this use the lower section, or gasoline chamber, shown in Fig. 1(*b*), shall be essentially the same as described in Paragraph (*b*) except that a ¼-in. needle valve shall be introduced in the coupling between the chambers. The volume of the gasoline chamber, including only the capacity enclosed by the valves, shall fulfill the requirements of Paragraph (*e*).

Section 3 (a).—Change the first sentence by the addition of the italicized words and figures, as follows:

The pressure gage shall be a Bourdon-type spring gage of test-gage quality 4½ to 5½ in. in diameter *provided with a nominal ¼-in. male-thread connection* with a passageway not less than $\frac{3}{16}$ in. in diameter from the Bourdon tube to the atmosphere.

Withdrawal of Tentative Standard:

Test for Neutralization Number of Petroleum Products and Lubricants (D 188 - 41 T)

Published as Information Only:

Test for Oil Content of Paraffin Wax, with editorial changes

Test for Color of Lubricating Oil by Means of Photoelectric Colorimeter, with editorial changes

Test for Potential Gum in Aviation Gasoline

Test for Oxidation Characteristics of Heavy-Duty Crankcase Oils, with editorial changes

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[a] Suggested corrosion inhibitors are sodium nitrite or triethanolamine phosphate.

Sectional Committee Z11 on Petroleum Products and Lubricants:

Report not preprinted presented from manuscript by T. A. Boyd, chairman.

Committee D-4 on Road and Paving Materials:

Report presented by J. E. Myers, chairman, and the following actions taken:

Accepted as Tentative:

Revisions of Spec. for Crushed Slag for Bituminous Macadam Base and Surface Courses (D 487 - 41 T), with the addition of the word "compacted" in the requirements for weight per cubic foot

Test for Abrasion of Gravel by Use of the Deval Machine (D 289 - 37 T)

Adopted as Standard:

Test for Modified Miscibility and Cement Mixing of Emulsified Asphalts (D 244 - 41 T), this recommendation, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee D-4,[8] the method to be incorporated in the Standard Methods of Testing Emulsified Asphalts (D 244 - 40), with the following revisions:

Section 6.—Change the second sentence to read as follows by the addition of the italicized word and figure and the omission of those in brackets:

> This test is not applicable [where 5] *when 2* per cent or more coagulation is anticipated.

Section 9 (d).—Change the first sentence to read as follows by the addition of the italicized words and figures and the omission of the words in brackets:

> Pour the mixture through the tared 1410-micron (No. 14) iron [wire] sieve *of approximately 3 in. diameter* and rinse [with water until the wash water is clear] *by pouring distilled water from a receptacle held at a height of approximately 6 in.*

Revision of Test for Determination of Bitumen (D 4 - 27), with editorial changes

Revision of Test for Proportion of Bitumen Soluble in Carbon Tetrachloride (D 165 - 27) with editorial changes

Editorial Changes Accepted:

Test for Softening Point of Tar Products (Cube-in-Water Method) (D 61 - 38)

Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (D 36 - 26)

Spec. for Materials for Stabilized Base Course (D 556 - 40 T)

Spec. for Materials for Stabilized Surface Course (D 557 - 40 T)

Testing Emulsified Asphalts (D 244 - 40)

Test for Distillation of Tar Products Suitable for Road Treatment (D 20 - 30), the following editorial changes, not preprinted, were accepted by unanimous consent:

Section 4 (b).—Change to read as follows by the addition of the italicized words:

> (*b*) The axis *of the bulb* of the flask through *the center* of the neck shall be vertical *and the thermometer shall be aligned on this axis.*

Section 4 (c).—Change the first sentence to read as follows by the addition of the italicized words and the omission of those in brackets:

> The distance from the [bulb of the thermometer] *neck of the flask* to the outlet end of the condenser tube shall be not more than 600 nor less than 500 mm.

Fig. 3.—Change the 500–600 mm. dimension to conform with the above revision in Section 4 (*c*) and as shown in Fig. 3 of the Standard Method of Test for Distillation of Creosote (D 246 - 39) but without change in position to the thermometer.

It was announced that the committee would have the following additional recommendations to present to the Society subsequent to the annual meeting through Committee E-10 on Standards:[9]

Proposed Tentative Standards:

Spec. for Crushed Gravel for Waterbound Base and Surface Courses

Spec. for Gravel for Bituminous Concrete Base and Surface Courses

Revisions of Tentative Specifications for:

Standard Sizes of Coarse Aggregate for Highway Construction (D 448 - 41 T)

Crushed Slag for Bituminous Concrete Base and Surface Courses (D 485 - 38 T)

Crushed Stone for Bituminous Concrete Base and Surface Courses (D 486 - 39 T)

Crushed Slag for Water-Bound Base and Surface Courses (D 488 - 38 T)

Crushed Stone for Water-Bound Base and Surface Courses (D 489 - 40 T)

Revision of Tentative Method of:

Sampling Stone, Slag, Gravel, Sand, and Stone Block for Use as Highway Materials (D 75 - 40 T)

It was also announced that Committee D-4 has referred to letter ballot the Proposed Method of Test for Moisture Density Relations of Soil originated by Committee D-18 on Soils for Engineering Purposes but coming under the joint jurisdiction of the two committees.[9]

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[8] Results of the letter ballot on this recommendation, indicating its approval, are given in the report of Committee D-4, see p. 350.

[9] See report of Committee D-4, Editorial Note, p. 350.

Committee D-18 on Soils for Engineering Purposes:

Report presented, in the absence of the chairman, by Wesley R. Nelson, secretary.

It was announced that the following recommendations would be presented to the Society through Committee E-10 on Standards, subsequent to the annual meeting:

Proposed Tentative Method of Test for Specific Gravity of Soils
Tentative Revision of Standard Method of Mechanical Analysis of Soils (D422 - 39)[10]
Revision of Tentative Methods of Surveying and Sampling Soils for Highway Subgrades (D 420 - 40 T)[10]

Sectional Committee A37 on Road and Paving Materials:

Progress report, not preprinted, presented from manuscript, in the absence of the chairman, by R. E. Hess, secretary.

Committee D-8 on Bituminous Waterproofing and Roofing Materials:

Report presented, in the absence of the chairman, by C. S. Reeve, and the following actions taken:

Accepted as Tentative:

Spec. for Coal-Tar Pitch for Steep Built-Up Roofs
Spec. for Asphalt-Saturated and Asphalt-Coated Asbestos Felt for Use in Constructing Built-Up Roofs
Revisions of Spec. for Asphalt for Damp-proofing and Waterproofing (D 449 - 37 T)
Revision of Spec. for Asphalt-Saturated Asbestos Felt for Use in Constructing Built-Up Roofs (D 250 - 27), and reversion to tentative

Adopted as Standard:

Spec. for Asphalt-Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (D 226 - 41 T)
Spec. for Woven Cotton Fabrics Saturated with Bituminous Substances for Use in Waterproofing (D 173 - 40 T)
Methods of Testing Films Deposited from Bituminous Emulsions (D 466 - 38 T)
Revision of Spec. for Coal-Tar Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (D 227 - 41), with the following additional revisions, accepted as an editorial change:

Section 3.—Change the last sentence to read as follows:

> The surface of the felt shall be uniformly smooth. Upon splitting or tearing on the bias, the felt shall appear reasonably free from lumps of underbeaten stock (that is, stock which has not been beaten or shredded into fiber in the process of manufacture) and particles of foreign substances (that is, fragments of stone, metal, leather, rubber, etc.).

Withdrawal of Tentative Standard:

Spec. for Asphalt Roofing Surfaced with Fine Mineral Granules (D 248 - 41 T)

Editorial Changes Accepted:

Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing (D 146 - 38 T)
Testing Asphalt Roll Roofing, Cap Sheets, and Shingles (D 228 - 37 T).
Spec. for Creosote for Priming Coat with Coal-Tar Pitch in Dampproofing and Waterproofing (D 43 - 41), an editorial change, not preprinted, providing for the insertion of the following as a footnote in connection with the requirement for consistency in Section 2:

> The creosote shall be rated as crystal free if no crystals are formed when 100 ml. of the sample are maintained at a temperature of 5 C. for 3 hr. in a 125-ml. Erlenmeyer flask with occasional stirring.

Spec. for Asphalt Roofing Surfaced with Powdered Talc or Mica (D 224 - 41 T)
Spec. for Asphalt Shingles Surfaced with Coarse Mineral Granules (D 225 - 41 T)
Spec. for Asphalt Roofing Surfaced with Coarse Mineral Granules (D 249 - 41 T)
Spec. for Wide Selvage Asphalt Roofing Surfaced with Coarse Mineral Granules (D 371 - 41 T)

Editorial changes, not preprinted, in the above four standards were accepted, as follows:

Section 3.—Change the last sentence to read as recommended above in Section 3 of Specifications D 227.

Section 4.—Change to read as follows by the addition of the italicized words and the omission of the word in brackets:

> 5. The ***bitumen of the*** saturant and coatings shall be composed [principally] of asphaltic materials.

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[10] See Report of Committee D-18, Editorial Note, p. 450.

Papers:

The following papers were presented:

Statistical Study of the Precision of Methods for Analysis of Coal and Coke—H. H. Lowry and Charles O. Junge, Jr., presented by Mr. Lowry.

An Experimental Investigation of the British Standard Method for the Crucible Swelling Test for Coal—H. N. Ostborg, H. R. Limbacher, and Ralph A. Sherman, presented by Mr. Ostborg.

A Rapid Method of Estimating the A.S.T.M Ring-and-Ball Softening Point of Asphalts—A. B. Hersberger and C. Overbeck, presented by Mr. Hersberger.

NINTH SESSION—STEEL, FERRO-ALLOYS, EFFECT OF TEMPERATURE

WEDNESDAY, JUNE 24, 1.30 P.M.

Session Chairman: Past-President H. H. Morgan.

Committee A-1 on Steel:

Report presented by N. L. Mochel, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Copper Brazed Steel Tubing, published as information in the preprinted report, accepted by unanimous consent, subject to favorable letter ballot of Committee A-1[11]

Proposed Spec. for Welded Alloyed Open Hearth Iron Pipe, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee A-1[11]

Revisions of:

Spec. for Light Gage Structural Quality Flat Hot-Rolled Carbon Steel (0.2499 and 0.1874 in. to 0.0478 in. in Thickness) (A 245 – 41 T), with the following additional changes accepted by unanimous consent, subject to favorable letter ballot of Committee A-1:[11]

Section 7 (*a*).—Insert in the table for the three grades A, B, and C, the following minimum values for elongation in 2 in. of 29, 25, and 22 per cent, respectively.

Section 9 (*b*).—Change from the form as preprinted to read as follows:

> (*b*) Tension test specimens shall have their longest axis in the direction of the last hot rolling and shall be the full thickness of the material as rolled. Test specimens may be machined to the form and dimensions shown in Fig. 1 or 2 as specified.

Figs. 1 and 2.—Add as Fig. 1 the standard rectangular test specimen with 8 in. gage length and as Fig. 2 the standard rectangular test specimen with 2 in. gage length. These are the same as Figs. 1 and 2 which appear in the Tentative Methods of Tension Testing of Metallic Materials (E 8 – 40 T).

Spec. for Light Gage Structural Quality Flat Rolled Carbon Steel (0.0477 to 0.0225 in. in Thickness) (A 246 – 41 T)

Spec. for Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F. (A 158 – 41 T), with the following additional changes accepted by unanimous consent, subject to favorable letter ballot of Committee A-1:[11]

Section 8 (*a*).—Revise the elongation requirements by changing the table from its present form to read as shown in the accompanying Table I.

Spec. for Seamless Carbon-Molybdenum Alloy Steel Pipe for Service at Temperatures from 750 to 1000 F. (A 206 – 41 T), with the following additional changes accepted by unanimous consent, subject to favorable letter ballot of Committee A-1:[11]

Section 2.—Delete Paragraphs (*d*) and (*e*) and add a new Paragraph (*d*) to read as follows:

> (*d*) When requested by the purchaser and so stated in the order, hot-finished pipe in the as-finished condition in nominal sizes 3 in. and over made to schedules 140 and 160 shall have a structural grain size which is predominately 3 to 6 as measured on an A.S.T.M. grain size chart.

Section 12.—Change from its present form to read as follows:

> 12. When requested by the purchaser and so stated in the order, the manufacturer shall furnish one photomicrograph at 100 diameters from a specimen of pipe in the as-finished condition for each individual size and wall thickness from each melt, for pipe in nominal sizes 3 in. and over made to schedules 140 and 160, to demonstrate that such pipe meets the requirements of Section 2(*d*) when the order states that those requirements are to be met. Such photomicrographs shall be suitably identified as to pipe size, wall thickness, and melt. No photomicrographs for the individual pieces purchased shall be required except as specified in Paragraph S-7.

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[11] Results of the letter ballot on this recommendation indicating its approval, are given in the report of Committee A-1, see p. 80.

Supplementary Requirements, Section S7.—Change from its present form to read as follows:

> S7. In addition to the photomicrographs which may be required under Section 12, for hot-finished pipe in nominal sizes 3 in and over made to schedules 140 and 160 and specified to meet the requirements of Section 2(*d*), the purchaser may specify that photomicrographs shall be furnished from each end of one or more pieces from each lot of pipe in the as-finished condition. The purchaser shall state in the order the number of pipes to be tested from each lot. When photomicrographs are required on each piece, the photomicrographs from each lot of pipe in the as-finished condition which may be required under Section 12 may be omitted. All photomicrographs required shall be properly identified as to heat number, size, and wall thickness of pipe from which the section was taken. Photomicrographs shall be further identified to permit association of each photomicrograph with the individual piece of pipe it represents.

TABLE I.

	Ferritic		Austenitic	
Tensile strength, min., psi	60 000		75 000	
Yield point, min., psi	30 000		30 000	
	Longitudinal	Transverse	Longitudinal	Transverse
Elongation, min., per cent:				
Basic minimum elongation for walls 5⁄16 in. and over in thickness, strip tests, and for all small sizes tested in full section	30	20	50	40
When standard round 2-in. gage length test specimen is used	22	14	35	25
For strip tests, a deduction for each 1⁄32 in. decrease in wall thickness below 5⁄16 in. from the basic minimum elongation of the following percentage	1.50[a]	1.00[a]	2.50[a]	2.00[a]

[a] The following table gives the calculated minimum values:

Wall Thickness, in.	Ferritic		Austenitic	
	Longitudinal	Transverse	Longitudinal	Transverse
5⁄16 (0.3125)	30.00	20.00	50.00	40.00
9⁄32 (0.281)	28.50	19.00	47.50	38.00
1⁄4 (0.250)	27.00	18.00	45.00	36.00
7⁄32 (0.2187)	25.50	....	42.50	....
3⁄16 (0.1875)	24.00	....	40.00	....
5⁄32 (0.156)	22.50	....	37.50	....
1⁄8 (0.125)	21.00	....	35.00	....
3⁄32 (0.0937)	19.50	....	32.50	....
1⁄16 (0.0625)	18.00	....	30.00	....

Section 8 (*a*).—Revise the elongation requirements by changing the table from its present form to read as shown in the accompanying Table II.

Spec. for Lap-Welded and Seamless Steel Pipe for High-Temperature Service (A 106 - 41); reverted to status of tentative with following additional changes accepted by unanimous consent, subject to favorable letter ballot of Committee A-1,[11] reversion to tentative being recommended in order that the elongation requirements, the addition of the acid bessemer process requirements for this steel, and the revised flattening test requirements which appeared in the report as preprinted will all be incorporated immediately in the tentative specifications, instead of being published as tentative revisions of a standard.

Table I.—Change footnote 5 as preprinted to read as follows:

> When acid-bessemer steel is furnished for grades A or B seamless pipe the phosphorus content shall be 0.11 max. per cent and the carbon content shall be 0.25 max. per cent.

Spec. for Carbon-Steel Castings Suitable for Fusion Welding for Service at Temperatures up to 850 F. (A 216 - 41 T); with the following changes, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee A-1:[11]

Section 5 (*a*).—Change the requirement for silicon in both grades from the present value of "0.50 max." to read "0.60 max." per cent.

Spec. for Alloy-Steel Castings Suitable for Fusion Welding for Service at Temperatures from 750 to 1100 F. (A 217 - 41 T); with the following changes, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee A-1:[11]

Section 5 (a).—Change the requirement for silcon in grades WC1 and WC2 from the present value of "0.50 max." to read "0.60 max."

Adopted as Standard:

Spec. for Low-Alloy Structural Steel (A 242 - 41 T)

Spec. for Carbon-Steel Forgings for General Industrial Use (A 235 - 40 T)

TABLE II.

Tensile strength, min., psi	55 000	
Yield point, min., psi	30 000	
	Longitudinal	Transverse
Elongation, min., per cent:		
Basic minimum elongation for walls 5/16 in. and over in thickness, strip tests, and for all small sizes tested in full section	30	20
When standard round 2-in. gage length test specimen is used	22	14
For strip tests, a deduction for each 1/32 in. decrease in wall thickness below 5/16 in. from the basic minimum elongation of the following percentage	1.50[a]	1.00[a]

[a] The following table gives the calculated minimum values:

Wall Thickness, in.	Longitudinal	Transverse
5/16 (0.3125)	30.00	20.00
9/32 (0.281)	28.50	19.00
1/4 (0.250)	27.00	18.00
7/32 (0.2187)	25.50	....
3/16 (0.1875)	24.00	....
5/32 (0.156)	22.50	....
1/8 (0.125)	21.00	....
3/32 (0.0937)	19.50	....
1/16 (0.0625)	18.00	....

Spec. for Carbon-Steel Forgings for Locomotives and Cars (A 236 - 40 T)

Spec. for Alloy-Steel Forgings for General Industrial Use (A 237 - 40 T)

Spec. for Alloy-Steel Forgings for Locomotives and Cars (A 238 - 41 T)

Spec. for Factory-Made Wrought Carbon-Steel and Carbon-Molybdenum-Steel Welding Fittings (A 234 - 40 T)

Spec. for Atomic-Hydrogen-Arc-Welded and Electric-Resistance-Welded Alloy-Steel Boiler and Superheater Tubes (A 249 - 41 T), as revised

Spec. for Seamless Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 209 - 41 T)

Spec. for Heat-Treated Wrought Steel Wheels (A 244 - 41 T)

The recommendation, not preprinted, for adoption of the three last named tentative standards was accepted by unanimous consent, subject to favorable letter ballot of Committee A-1[11]

Revisions of:

Spec. for Chromium-Vanadium Steel Bars for Springs (A 60 - 39)

Spec. for Seamless Cold-Drawn Low-Carbon Steel Heat-Exchanger and Condenser Tubes (A 179 - 40)

Spec. for Electric-Resistance-Welded Steel Heat-Exchanger and Condenser Tubes (A 214 - 40)

Spec. for Seamless Alloy-Steel Boiler and Superheater Tubes (A 213 - 40)

Spec. for Carbon-Steel Bars for Springs (A 14 - 39); the following changes, not preprinted, were accepted by unanimous consent, subject to favorable letter ballot of Committee A-1:[11]

Section 1.—Revise to provide for addition of new Grade C, indicating that this is for applications where definitely specified.

Section 3.—Add the following chemical composition requirements for a new Grade C:

	Grade C
Carbon, per cent	0.85 to 1.05
Manganese, per cent	0.25 to 0.50
Phosphorus, max., per cent	0.05
Sulfur, max., per cent	0.05

Concurrent with this action was the withdrawal of the Standard Specifications for Carbon-Steel Bars for Vehicle and General Purpose Springs (A 58 - 27). Grade C is being added to Specifications A 14 to provide for a composition that was formerly covered by Specifications A 58.

Spec. for Welded and Seamless Steel Pipe (A 53 - 40); with the following additional changes, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee A-1:[11]

Section 1.—Add a new paragraph to read as follows:

This material is suitable for fusion welding.

Section 4.—Change this section from its present form to read as follows by the addition of the italicized words:

> 4. An analysis of two pipes from each lot of 500 lengths or fraction thereof may be made by the purchaser. Drillings for analysis shall be taken from several points around each pipe selected for analysis. The phosphorus content *of open-hearth or electric-furnace steel* thus determined shall not exceed that specified in Section 3 by more than 25 per cent. *For acid-bessemer seamless steel pipe, the phosphorus content shall not exceed the maximum specified in Section 3.*

The proposed tentative revisions in Sections 1 (*a*), 2, 3, 7, 10 (*b*), 17 and Table I, as preprinted were recommended for immediate adoption and accepted by unanimous action subject to favorable letter ballot of Committee A-1[11]

Spec. for Black and Hot-Dipped Zinc-Coated (Galvanized) Welded and Seamless Steel Pipe for Ordinary Uses (A 120 – 40)
Spec. for Molybdenum-Steel Plates for Boilers and Other Pressure Vessels (A 204 – 39)
Spec. for Carbon-Steel Plates for Stationary Boilers and Other Pressure Vessels (A 70 – 39)
Spec. for Low-Carbon Nickel-Steel Plates for Boilers and Other Pressure Vessels (A 203 – 39)
Spec. for Commercial Quality Hot-Rolled Bar Steels (A 107 – 40)
Spec. for Alloy-Steel Castings for Valves Flanges, and Fittings for Service at Temperatures from 750 to 1100 F. (A 157 – 41), with the following additional changes, accepted by unanimous consent, subject to favorable letter ballot of Committee A-1:[11]

Table I.—Change the carbon content of grades C 3A, C 3B, C 5A, and C 5B from "0.35 max. per cent" in the first two grades and "0.15 to 0.35 per cent" in the last two grades to read "0.30 max. per cent" in all four grades.

Table II.—Change the minimum tensile strength of grades C 5A and C 5B from "100,000 psi." to read "90,000 psi." and the minimum yield point from "65,000 psi." to read "60,000 psi."

Spec. for Steel for Bridges and Buildings (A 7 – 39)
Spec. for Structural Steel for Locomotives and Cars (A 113 – 39)
Spec. for Carbon-Steel Castings for Miscellaneous Industrial Uses (A 27 – 39)
Spec. for Carbon-Steel and Alloy-Steel Castings for Railroads (A 87 – 36)
Spec. for Alloy-Steel Castings for Structural Purposes (A 148 – 36)
Spec. for Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes (A 83 – 40)
Spec. for Electric-Fusion-Welded Steel Pipe (Sizes 30 in. and Over) (A 134 – 39)
Spec. for Electric-Resistance-Welded Steel Pipe (A 135 – 34)
Spec. for Electric-Fusion-Welded Steel Pipe (Sizes 8 in. to but not Including 30 in.) (A 139 – 39)
Spec. for Forged or Rolled Steel Pipe Flanges for General Service (A 181 – 37)
Spec. for Seamless Cold-Drawn Alloy-Steel (4 to 6 per cent Chromium) Heat-Exchanger and Condenser Tubes (A 187 – 40)
Spec. for Boiler and Firebox Steel for Locomotives (A 30 – 39)
Spec. for Electric-Fusion-Welded Steel Pipe for for High-Temperature and High-Pressure Service (A 155 – 36)

Withdrawal of Standards:

Spec. for Lock-Bar Steel Pipe (A 137 – 34)
Spec. for Carbon-Steel Bars for Vehicle and General Purpose Springs (A 58 – 27)

Reaffirmation of Standards:

Spec. for Open-Hearth Steel Girder Rails of Plain, Grooved, and Guard Types (A 2 – 27)
Spec. for Low-Carbon Steel Joint Bars (A 3 – 33)
Spec. for Medium-Carbon Steel Joint Bars (A 4 – 14)
Spec. for Soft Steel Track Spikes (A 65 – 33)
Spec. for Steel Screw Spikes (A 66 – 33)
Spec. for Steel Tie Plates (A 67 – 33)
Spec. for Low-Carbon Steel Track Bolts and Nuts (A 76 – 33)
Spec. for Rail-Steel Bars for Concrete Reinforcement (A 16 – 35)
Spec. for Cold-Drawn Steel Wire for Concrete Reinforcement (A 82 – 34)
Spec. for Axle-Steel Bars for Concrete Reinforcement (A 160 – 39)
Spec. for Fabricated Steel Bar or Rod Mats for Concrete Reinforcement (A 184 – 37)
Spec. for Welded Steel Wire Fabric for Concrete Reinforcement (A 185 – 37)
Spec. for Carbon-Steel Axles for Cars and Tenders (A 21 – 36)
Spec. for Austenitic Manganese-Steel Castings (A 128 – 33)
Spec. for Forge-Welded Steel Pipe (A 136 – 34)
Spec. for Riveted Steel and Wrought-Iron Pipe (A 138 – 34)

Committee A-9 on Ferro-Alloys:

Report presented, in the absence of the chairman, by C. M. Loeb, Jr., secretary, and the following action taken:

Adopted as Standard:

Spec. for Ferrochromium (A 101 - 39 T)

Sectional Committee B36 on Standardization of Dimensions and Materials of Wrought-Iron and Wrought-Steel Pipe and Tubing:

Progress report, not preprinted, presented from manuscript by H. H. Morgan, chairman.

Joint Research Committee on Effect of Temperature on the Properties of Metals:

Progress report, presented by N. L. Mochel, chairman.

Joint Committee on Definitions of Terms Relating to Heat Treatment:

Progress report, not preprinted, presented from manuscript by H. S. Rawdon, chairman.

Committee A-6 on Magnetic Properties:

Report presented by Thomas Spooner, chairman, and the following action taken:

Adopted as Standard:

Test for Magnetic Properties of Iron and Steel (A 34 - 41)

It was announced that the three following methods, which appeared in the preprinted report as information would be presented to the Society subsequent to the annual meeting for publication as tentative through Committee E-10 on Standards:[12]

Test for Permeability of Feebly Magnetic Materials

Test for Measuring Permeability and Core Loss of Flat-Rolled Magnetic Materials Using 28-cm. Specimen

Test for Incremental Permeability and Core Loss of Flat-Rolled Magnetic Materials at Low Alternating Inductions Using 28-cm. Specimen

Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys:

Report, not preprinted, presented from manuscript by Jerome Strauss, chairman, and the following actions taken:

Adopted as Standard:

Revisions of:

Spec. for Corrosion-Resisting Chromium-Nickel Steel Sheet, Strip, and Plate (A 167 - 41)

Spec. for Corrosion-Resisting Chromium Steel Sheet, Strip, and Plate (A 176 - 41)

Spec. for Corrosion-Resisting Chromium-Nickel Steel Sheet, Strip, and Plate for Fusion-Welded Unfired Pressure Vessels (A 240 - 41)

In the above three specifications the revisions, not preprinted, comprise changes in chemical composition requirements proposed in order to bring the specifications into agreement with practice and in accord with the current type classifications of the American Iron and Steel Institute.

Papers:

The following papers were presented:

The Physical Properties of Steel Wire as Affected by Variations in the Drawing Operations—Howard J. Godfrey, presented by the author.

Comparative Quality of Converter Cast Steel—C. E. Sims and F. B. Dahle, presented by Mr. Sims.

The Absolute Measurement of the Energy Loss in a Single Strip of Sheet Iron or Steel Under Alternating Magnetization—W. E. Shenk, presented by the author.

TENTH SESSION—MARBURG LECTURE

WEDNESDAY, JUNE 24, 4.30 P.M.

Session Chairman: President G. E. F. Lundell.

In introducing the Seventeenth Edgar Marburg Lecturer, President Lundell explained briefly the purpose of this annual memorial lecture, established 17 years ago as a means of honoring and perpetuating the memory of the first Secretary-Treasurer of the Society, Edgar Marburg thus commemorating one who contributed so much to the A.S.T.M. during the years of its early development. It brings to the Society membership valuable expositions by authorities in the sciences and in engineering concerning important developments in the field of engineering materials.

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[12] See report of Committee A-6, Editorial Note, p. 126.

He then presented Dr. Graham Edgar, Director of Research, Ethyl Corp., who had chosen as the subject of his lecture "Gasoline—Past, Present and Future." Doctor Edgar discussed at some length those properties of gasoline that have the greatest bearing on its suitability for use in internal combustion engines: namely, its volatility, distillation, vapor pressure, and knocking characteristics. Since the tendency of gasoline to knock is probably its most important single property, Doctor Edgar pointed out that this has been one of the most difficult problems and he presented a summary of the best technical opinions concerning the available tests, including the A.S.T.M.-C.F.R. method. Concerning gasoline chemistry he referred to the importance of research in this field which in large measure was a result of the knock test studies since this is related directly to chemical compositions. He mentioned that present knowledge of hydrocarbons has led to advances in many fields other than gasoline. In reviewing the current gasoline situation he also discussed the important part that gasoline will probably play in the future in air transportation. In conclusion, he stressed the very close relation between the petroleum and the automotive manufacturer as inseparable parts of the transportation industry, which parts must progress simultaneously in order for the public to receive the most efficient, economical, comfortable, and safer transportation which appears to be both technically and economically possible.

President Lundell, in expressing appreciation to Doctor Edgar for his fine exposition on a subject of particular interest to the A.S.T.M., presented to him on behalf of the Society, the Edgar Marburg Lecture Certificate.

ELEVENTH SESSION—ADDRESSES—PRESIDENT LUNDELL AND BRIGADIER-GENERAL C. D. YOUNG; AWARD OF DUDLEY MEDAL

WEDNESDAY, JUNE 24, 8.15 P.M.

Session Chairman: Past-President T. G. Delbridge

Committee E-10 on Standards:

Report presented by J. R. Townsend, chairman.

Committee E-9 on Research:

Report presented by G. F. Jenks, chairman.

Executive Committee:

This report, presented by Secretary-Treasurer C. L. Warwick, discussed the nature and extent to which the Society was contributing to the war effort. Reference was made to the procedure established by the Executive Committee for handling emergency standards and emergency alternate provisions in standards, which has resulted in the publication of six emergency specifications and test methods, 50 emergency alternate provisions, and one emergency alternate specification. The report also discussed briefly various requests received by A.S.T.M. from the War Production Board and other Government agencies in connection with problems relating to materials essential to the war effort. The report also covered general Society activities, membership, publications, finances, administrative matters relating to committee activities, and cooperative activities with other engineering societies.

The report announced that the Executive Committee had approved a change in the Regulations Governing Standing Committees, which would permit committees to submit to letter ballot recommendations dealing with standards without requiring that such matters be first considered at a meeting of the committee, this change in the regulations to become effective at the close of this annual meeting.

Address by General Young:

The chairman then introduced Brigadier-General Charles D. Young, Director of Procurement and Distribution, Services of Supply, U. S. A.; and Past-President of the Society, who presented a talk on "Organization of the Services of Supply of the War Dept." General Young spoke informally and off the record concerning many problems confronting the services of supply, one of the three major army components, the other two being the ground forces and the air forces. He referred to the tremendous distribution problem involved in the work of this branch of the service and stated that remarkable progress has been made in reducing the time from completion of production to delivery at the point of use. He stated that production was considerably ahead of the designed capacity and that this was due to advanced planning and a number of stimulating causes. General Young stressed the need for stimulating research on synthetic rubber and referred to the possible future use of various substitutes for alloys requiring nickel, chromium, tungsten, vanadium, and related metals. He also stressed the importance of steel as a substitute for copper in certain ordnance materials and stated that the authorized expenditure of $24,000,000,000 gave some idea of the tremendous production in this one branch of the Army.

Presidential Address:

The chairman then presented the President, Dr. G. E. F. Lundell, who delivered the presidential address on "Chemical Requirements and Chemical Analysis." Doctor Lundell's address was based on his 25 years of experience in this field of materials testing. He indicated that the operations of a chemical analysis can be divided into those that make the determination possible and those which attend the determination itself. He indicated that the former is more important in applied analysis and that it would be helpful to the understanding of analytical chemistry if the term chemical analysis were applied to true analysis. He paid tribute to the pioneering work in this field of the Society's first president, Dr. Charles B. Dudley. A considerable part of the address was devoted to a discussion and explanation of precision and accuracy, and examples of how incongruous some requirements appear when consideration is given to the personal equation and apparatus limitations. The importance and increasing use of spectrographic procedures in quantitative and qualitative analysis in the inorganic field, and of microanalysis in the organic field received special mention. In conclusion he referred to the improvements made by chemists in shortening the time required for chemical analysis which has made quite a contribution in the evolution of current manufacturing processes.

Award of Dudley Medal:

The chairman presented, as the recipients of the award of the Charles B. Dudley Medal, Francis C. Todd, Assistant Professor, Petroleum and Natural Gas Engineering, and A. W. Gauger, Director, Mineral Industries Research, The Pennsylvania State College, for their paper on "Studies on the Measurement of Water Vapor in Gases," presented at the 1941 Annual Meeting of the Society. The Dudley Medal is awarded annually by the Society to the author or authors of a technical paper considered to be of outstanding merit, and constituting an original contribution on research in engineering materials. In thus recognizing meritorious contributions in the materials field, the medal commemorates the name of the first President of the Society, Charles B. Dudley.

On behalf of and as the representative of the Society, President Lundell then made the sixteenth award of the Charles B. Dudley Medal to Messrs. Todd and Gauger.

In expressing appreciation of the honor conferred upon the authors by this award Mr. Gauger made the following remarks:

> I realize that the hour is late, but I think that Professor Todd and I would be churlish if we did not express our very, very deep appreciation of this award. I wish to thank the committee and the members of this Society on behalf of my colleague and myself.
>
> There are one or two other things that I should mention, because they will be of interest to you, I know. We are deeply grateful also because this work is part of the work of a relatively new committee, Committee D-3 on Gaseous Fuels, and so, on behalf of my friends on the committee, who, to paraphrase a common statement, have been our best friends and also our severest critics, I want to thank the Society.
>
> I wish also to publicly express our appreciation at this time for a grant-in-aid which we received from the Research Committee of the Society several years ago, amounting to two hundred dollars. We are deeply grateful for that because it enabled us to get our second wind and finish our apparatus when we were broke.

Recognition of Forty-Year Members:

The chairman announced that in accordance with action taken by the Executive Committee, certificates were being awarded this year to nine individuals and six companies who had attained 40 years of membership in the Society. Presentation of the certificates was then made by the Secretary-Treasurer to the following:

Charles Derleth, Jr., University of California.
W. C. Du Comb, W. C. Du Comb Co., Inc.
W. R. Edwards, Baltimore, Md.
J. E. Greiner, The J. E. Greiner Co.
N. F. Harriman, Procurement Division, Treasury Dept.
W. M. Saunders, Analytical and Consulting Chemist, Providence, R. I.
H. E. Smith, Materials Engineer, White Plains, N. Y.
Bradley Stoughton, Lehigh University.
J. L. Van Ornum, Washington University.
Ajax Metal Co., Philadelphia, Pa.
International Harvester Co., Chicago, Ill.
Jones & Laughlin Steel Co., Pittsburgh, Pa.
Lukens Steel Co., Coatesville, Pa.
Sherwin-Williams Co., Cleveland, Ohio.
Standard Steel Works Division, The Baldwin Locomotive Works, Burnham, Pa.

Election of Officers:

The chairman then recognized C. S. Reeve, chairman of the Committee of Tellers, who reported results of the letter ballot on election of officers. Of the 1313 ballots cast, the results were as follows:

For President, to serve for 1 yr.:
H. J. Ball, 1313 votes.
For Vice-President, to serve for 2 yr.:
P. H. Bates, 1312 votes.
For members of the Executive Committee, to serve for 2 yr.:
R. P. Anderson, 1313 votes.
M. H. Bigelow, 1313 votes.
J. H. Foote, 1311 votes.
Alexander Foster, Jr., 1312 votes.
L. H. Fry, 1313 votes.

The chairman then introduced the newly elected members of the Executive Committee, requesting that they rise as their names were called.

At the request of the chairman, Past-President G. H. Clamer and J. L. Miner, escorted to the chair the Vice-President-Elect, P. H. Bates, who expressed appreciation of the honor of his election.

The chairman then requested Past-President H. H. Morgan and Vice-President Dean Harvey to escort to the chair the President-Elect, H. J. Ball, who spoke as follows in acknowledging the honor of his election:

> Your election of me as your President for the coming year I deem to be an award of high honor. I hereby acknowledge it with deep appreciation.
>
> I should also like to share that honor, if it were possible, with every member of Committee D-13 on Textile Materials, a group of fine and loyal men and women with whom it has been a real pleasure to work and who, I feel, are sharing with me at this time the pleasure of this occasion.
>
> We all realize that the year into which we are passing is one whose effects upon the life, activities, and plans of the A.S.T.M. and of its members are difficult to estimate or predict. The main issue of the moment is to assist in every way possible any effort which will help in any degree to bring this war to an early and successful conclusion.
>
> You have already learned that the officers and the Executive Committee of the Society have been giving and will continue to give their best thought to plans which will make the influence of the A.S.T.M. most effective for this purpose. I take this opportunity to express recognition of the valuable work and service which has been rendered by hundreds of our members toward this same goal, and to ask for their continued support throughout the coming year.

TWELFTH SESSION—METHODS OF TESTING, CORROSION, FATIGUE

THURSDAY, JUNE 25, 9.30 A.M.

Session Co-Chairmen: Carl D. Hocker; Jerome Strauss

Committee E-8 on Nomenclature and Definitions:

Report presented by Cloyd M. Chapman, chairman, and the following action taken:

Adopted as Standard:

Def. of the Term Screen (Sieve) (E 13 – 28 T), as revised

It was announced that the Committee had approved for adoption as standard the Tentative Definitions of Terms Relating to Refractories (C 71 – 40 T), proposed by Committee C-8 on Refractories.

It was also announced that the Tentative Definitions of Terms Relating to Rheological Properties of Matter (E 24 – 40 T), being recommended for adoption as standard this year by Committee E-1 on Methods of Testing, had been circularized in the committee and that several suggestions resulting from this review would be discussed with Committee E-1 during the year.

The recommendation for adoption as standard of the revised definition for perforated gypsum lath, as proposed by Committee C-11 on Gypsum, was reviewed by Committee E-8 and action taken to request this committee to defer formal adoption of this definition for a year in order to provide opportunity for the discussion of desirable changes.

Committee E-1 on Methods of Testing:

Report presented by W. H. Fulweiler, chairman, and the following actions taken:

Accepted as Tentative:

Revisions of Spec. for A.S.T.M. Theromometers (E 1 – 41 T), kinematic viscosity thermometers

Adopted as Standard:

Hardness Conversion Table for Cartridge Brass

Test for Rockwell Hardness of Metallic Materials (E 18 – 39 T)

Def. of Terms Relating to Rheological Properties of Matter (E 24 – 40 T)

Adopted as Standard:

- Spec. for A.S.T.M. Thermometers (E 1 - 41 T):
  - Turpentine Distillation Thermometer E-1 (27C - 40T), replacing Standard Thermometer E-1 (27C - 39)
  - Titer Test Thermometer E-1 (36C - 41T)
- Tension Testing of Metallic Materials (E 8 - 40 T), as revised, with the following additional editorial changes, not preprinted, accepted by unanimous consent:

  Section 5.—To correct a typographical error, change the figure "0.0005 in." to read "0.005 in." in the revision of this section as preprinted.

  Section 18.—Add to this section which covers the speed of testing requirements an additional sentence reading as follows:

  > When the tensile properties of a material are appreciably affected by speed of straining suitable limits for rate of straining should be included in the purchase specifications covering that material.[a]

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  [a] This is the responsibility of those preparing the purchase specifications.

  Change Paragraph 18 (*c*) to read as follows by the addition of the italicized word and the omission of those in brackets:

  > (*c*) The change in tensile strength values produced by a change in the rate of straining is [usually considerably] *often* less than that observed for the yield strength. The range of permissible speeds is, therefore, wider.

- Revisions of Spec. for A.S.T.M. Thermometers (E 1 - 39):
  - High Distillation Thermometers E-1 (8C - 39) and E-1 (8F - 39), with a further change from the form as preprinted providing for the omission of the explanatory footnote and changing the distance from the bottom of the bulb to the graduation line at 0 C. (32 F.) from "25 to 35 mm. (0.98 to 1.38 in.)" to read "25 to 45 mm. (0.98 to 1.77 in.)." This change was accepted by unanimous consent subject to favorable letter ballot by Committee E-1.[13]
  - Open Flash Thermometers E-1 (11C - 39) and E-1 (11F - 39)
  - Thermometer for Stability Test of Soluble Nitrocellulose E-1 (26C - 39).

It was announced that the recommendation in the preprinted report for adoption as standard of the Specifications for the Tung Oil Thermometer E-1 (32 C - 39 T) had been withdrawn by the committee.

Mr. Fulweiler then called upon H. F. Moore to describe the activities of Technical Committee I on Mechanical Testing, and on E. C. Bingham to report on the work of Technical Committee II on Consistency, Plasticity, and Related Properties.

The paper by J. P. Bader and E. L. Peffer on "Methods of Testing Hydrometers," to be appended to the report, was presented from manuscript by W. H. Watson.

Committee A-5 on Corrosion of Iron and Steel:

Report presented, in the absence of the chairman, by C. D. Hocker.

Research Committee on Fatigue of Metals:

Report presented by H. F. Moore, chairman, who also summarized the paper by H. F. Moore and D. Morkovin entitled "Progress Report on the Effect of Size of Specimen on Fatigue Strength of Three Types of Steel," appended to the report.

Committee E-4 on Metallography:

Progress report, not preprinted, was presented from manuscript by L. L. Wyman, chairman.

Papers:

The following papers were presented:

- Hardness Conversion relationships—Robert H. Heyer, presented by the author.
- A Scanning Electron Microscope[14]—V. K. Zworykin, J. Hillier and R. L. Snyder, presented from manuscript by Mr. Hillier.
- Hydraulically - Supported Spherically - Seated Compression Testing Machine Platens—R. L. Templin, presented by the author.
- The Influence of the Combination of Principal Stresses in Fatigue of Metals—D. J. McAdam, Jr., presented by the author.
- Evaluation of Fatigue Damage of Steel by Supplementary Tension-Impact Tests—J. A. Kies and W. L. Holshouser, presented by Mr. Kies.
- Fatigue Tests as a Means of Evaluating Corrosion Damage of Sheet Metals—W. H. Mutchler and J. A. Kies, presented by Mr. Mutchler.

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[13] Results of the letter ballot on this recommendation indicating its approval appear in the report of Committee E-1, see p. 458.

[14] Published in ASTM BULLETIN, No. 117, August, 1942, p. 15.

THIRTEENTH SESSION—NON-FERROUS METALS

THURSDAY, JUNE 25, 2 P.M.

Session Co-Chairmen: President G. E. F. Lundell; Vice-President Dean Harvey

Committee B-8 on Electrodeposited Metallic Coatings:

Report presented by E. M. Baker, chairman.

Committee B-7 on Light Metals and Alloys, Cast and Wrought:

Report presented by Sam Tour, chairman, and the following actions taken:

Accepted as Tentative:

Revisions of Spec. for Aluminum-Base Alloy Sand Castings (B 26 – 41 T)

Revisions of Spec. for Aluminum for Use in Iron and Steel Manufacture (B 37 – 41 T), with the following additions, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee B-7:[15]

Table I.—Change from its present form to read as follows:

Grade	Aluminum, min. per cent by difference	Copper, max., per cent	Zinc, max., per cent	Magnesium, max., per cent	Total of All Impurities,[a] Including Iron, Silicon, Manganese, max., per cent
98	98.0	0.2	0.2	0.5	2.0
94 to 96	94.0	4.0	1.0	1.0	6.0
90 to 94	90.0	6.5	2.0	2.0	10.0
82.5 to 90	82.5	8.0	2.5	2.5	17.5

[a] Commercial grades of aluminum may also contain small amounts of one or more of the following elements: nickel, lead, tin, bismuth, chromium, molybdenum, and titanium. These minor elements need not be determined chemically or spectrographically unless otherwise specified in the contract or order.

Revisions of Spec. for Aluminum-Base Alloys in Ingot Form for Sand Castings (B 58 – 41 T), with the addition of the following requirements for new alloy ZG 41 (formerly alloy O), not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee B-7:[15]

Table I.—Add a new alloy ZG 41 (formerly alloy O) conforming to the following requirements as to chemical composition:

Copper, max., per cent	0.2
Iron, max., per cent	0.8
Silicon, max., per cent	0.3
Magnesium, per cent	0.5 ± 0.1
Zinc, per cent	$5.0\left\{\begin{matrix}+0.7\\-0.2\end{matrix}\right.$
Titanium, per cent	0.2 ± 0.1
Chromium, per cent	0.5 ± 0.1
Total other impurities, each max., per cent	0.05
Aluminum	remainder

Revisions of Spec. for Aluminum-Base Alloys in Ingot Form for Die Castings (B 125 – 39 T), with the following changes, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee B-7:[15]

Table I.—Change Alloy VII to conform to the following requirements:

	Alloy VII
Copper, per cent	3.5 to 4.5
Silicon, per cent	4.5 to 7.5
Nickel, max., per cent	0.3
Iron, max., per cent	1.0
Zinc, max., per cent	0.5
Manganese, max., per cent	0.5
Magnesium, max., per cent	0.10
Total other impurities, max., per cent	0.5
Single impurity not specifically mentioned, max., per cent	0.15
Aluminum	remainder

Revisions as given below in the following six specifications, not preprinted, were accepted by unanimous consent, subject to favorable letter ballot of Committee B-7:[15]

Spec. for Aluminum Sheet and Plate (B 25 – 41 T)

Aluminum-Alloy (Duralumin) Sheet and Plate (Aluminum - Copper - Magnesium - Manganese) (B 78 – 41 T)

Aluminum-Manganese Alloy Sheet and Plate (B 79 – 41 T)

Aluminum-Alloy (Duralumin Bars, Rods, Wire and Shapes (Aluminum-Copper-Magnesium-Manganese) (B 89 – 41 T)

Aluminum-Magnesium-Chromium Alloy Sheet and Plate (B 109 – 41 T)

Aluminum-Manganese Alloy Sheet and Plate for Use in Welded Pressure Vessels (B 126 – 40 T)

Section 3.—Add the following requirement for permissible zinc content in the tabulation of chemical composition requirements:

Zinc, max., per cent........................0.10

Change the requirement for other elements from its present form to read as follows:

Other elements, max., per cent........ $\left\{\begin{matrix}\text{each..0.05}\\\text{total..0.15}\end{matrix}\right.$

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[15] Results of the letter ballot on this recommendation, indicating its approval appear in the report of Committee B-7, see p. 221.

The following code system devised for classification of designations of aluminum base alloys was accepted for publication in the report of Committee B-7:

1. One or two letters will be used to designate the major alloying elements. If there is but one major alloying element, only one letter will be used. If there are two major alloying elements present, two letters will be used, the first letter designating that element present in the larger amount.

2. Numbers will be used following the letters which further define the number of alloying elements. If this number is below 20, it will indicate that only those elements designated by the code letters are present in the alloy. If this number is between 20 and 40, it will indicate that there is one alloying element present other than those indicated by the code letters. If the number is between 40 and 60, it will indicate that there are two alloying elements present other than those indicated by the code letters. Similarly, a number between 60 and 80 will indicate three additional alloying elements and so on.

3. Only those elements are considered alloying elements that are intentionally added and are therefore defined by both minimum and maximum limits in the specification.

4. The code letters to be used are as follows:

A—aluminum
C—copper
G—magnesium
M—manganese
N—nickel
R—chromium
S—silicon
Z—zinc

The use of the letter "A" for aluminum is necessary since various grades and purities are available in both ingot and wrought forms.

In order to provide for the application of the above code system to the various aluminum alloys under the jurisdiction of Committee B-7, action was taken approving editorial changes in the specifications to incorporate appropriate code letters for the alloys covered.

Committee B-6 on Die-Cast Metals and Alloys:

Report presented by J. R. Townsend, chairman, and the following actions taken:

Adopted as Standard:

Spec. for Aluminum-Base Alloy Die Castings (B 85 - 39 T), with the following revision, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee B-6:[15a]

Table I.—Replace alloy No. VII by new alloys VIIA and VIIB conforming to the following requirements as to chemical composition:

	Alloy VIIA	Alloy VIIB
Copper, per cent	3.5 to 4.5	3.5 to 4.5
Silicon, per cent	4.5 to 7.5	4.5 to 7.5
Nickel, max., per cent	0.3	0.3
Iron, max., per cent	2.0	1.3
Zinc, max., per cent	1.0	0.6
Manganese, max., per cent	0.5	0.5
Magnesium, max., per cent	0.10	0.10
Total other impurities, max., per cent	0.5	0.5
Any one impurity, max., per cent	0.15	0.15
Aluminum	remainder	remainder

The paper by E. H. Kelton on "Fatigue Testing of Zinc-Base Alloy Die Castings," to be appended to the report, was presented by the author.

Committee B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys:

Report presented by Dean Harvey, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Round Nickel Wire for Lamps and Electronic Devices

Revision of Test for Nickel and Nickel-Alloy Wire and Ribbon for Electronic Tube Filaments (B 118 - 39 T)

Revision of Test for Lateral Wire for Grids of Electronic Devices (B 156 - 41 T)

Adopted as Standard:

Test for Sleeves and Tubing for Radio Tube Cathodes (B 128 - 40 T)

Reaffirmation of Standards:

Test for Resistivity of Metallic Materials (B 63 - 36)

Test for Thermoelectric Power of Electrical Resistance Alloys (B 77 - 33)

Test for Temperature-Resistance Constants of Alloy Wires for Precision Resistors (B 84 - 36)

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys:

Report presented by Sam Tour, chairman.

[15a] Results of the letter ballot on this recommendation indicating its approval appear in the report of Committee B-6, see p. 216.

Committee B-5 on Copper and Copper Alloys, Cast and Wrought:

Report presented, in the absence of the chairman, by J. J. Kanter second vice-chairman, and the following actions taken:

Accepted as Tentative:

Revisions of Spec. for Naval Brass Rods (B 21 - 40 T)

Revisions of Spec. for Copper-Base Alloys in Ingot Form for Sand Castings (B 30 - 41 T)

Revisions of Spec. for Seamless Copper Tubing, Bright Annealed (B 68 - 41 T)

Revisions of Spec. for Copper-Base Alloy Forging Rods, Bars, and Shapes (B 124 - 41 T), with the following further changes in the proposed requirements for alloy No. 2, accepted by unanimous consent, subject to favorable letter ballot, of Committee B-5:[16]

Table I.—Change the chemical composition limits of alloy No. 2 by deleting the present specific maximum requirements for nickel and tin, increasing the requirement for iron to 0.30 per cent, max., and reducing the requirement for the total of the named elements from "99.90" per cent to read "99.50" min., per cent

Revisions of Spec. for Copper Rods, Bars, and Shapes (B 133 - 40 T)

Revisions of Spec. for Brass Wire (B 134 - 41 T)

Revisions of Spec. for Miscellaneous Brass Tubes (B 135 - 40 T)

Revisions of Spec. for Phosphor Bronze Bars, Rods, and Shapes (B 139 - 41 T)

Revisions of Spec. for Tin-Bronze and Leaded Tin-Bronze Sand Castings (B 143 - 41 T)

Revisions of Spec. for High-Leaded Tin-Bronze Sand Castings (B 144 - 41 T)

Revisions of Spec. for Leaded Red Brass and Leaded Semi Red Brass Sand Castings (B 145 - 41 T)

Revisions of Spec. for Leaded Yellow Brass Sand Castings for General Purposes (B 146 - 41 T)

Revisions of Spec. for High-Strength Yellow Brass and High-Strength Leaded Yellow Brass Sand Castings (B 147 - 41 T)

Revisions of Spec. for Aluminum-Bronze Sand Castings (B 148 - 41 T)

Revisions of Spec. for Leaded Nickel-Brass (Leaded Nickel-Silver) and Leaded Nickel-Bronze (Leaded Nickel-Silver) Sand Castings (B 149 - 41 T)

Revisions of Spec. for Copper-Nickel-Zinc Alloy Rods and Wire (B 151 - 41 T)

Revisions of Spec. for Phosphor Bronze Wire (B 159 - 41 T)

Revisions of Spec. for Cartridge Brass Sheet, Strip, and Disks (B 19 - 41 T), with the following changes, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee B-5:[16]

Section 14.—Change the second sentence to read as follows by the addition of the italicized word and the omission of the words in brackets:

> The tolerances for sheet *and* strip [and disks] shall be those shown in Table V.

Section 16.—Change the table of diameter tolerances to agree with Army Specifications 57-173-B.

Adopted as Standard:

Spec. for Copper-Silicon Alloy Plate and Sheet for Pressure Vessels (B 96 - 41 T)

Spec. for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 - 40 T)

Revisions of Spec. for Copper Bars for Locomotive Staybolts (B 12 - 33)

Revisions of Spec. for Free-Cutting Brass Rod for Use in Screw Machines (B 16 - 41) with the following change, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee B-5:[16]

Section 4.—In the table of chemical composition requirements, change the fourth item to read as follows by the addition of the italicized word:

> Materials other than copper, lead, *iron*, and zinc, max., per cent.................................0.50

Revisions of Spec. for Brass Pipe, Standard Sizes (B 43 - 41)

Revisions of Spec. for Steam or Valve Bronze Castings (B 61 - 41), with the following further change, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee B-5:[16]

Section 4.—Change the chemical composition requirements to read as follows:

	Min.	Max.
Copper, per cent	86.50	....
Tin, per cent	5.50	6.50
Lead, per cent	1.00	2.00
Zinc, per cent	2.50	5.00
Nickel, per cent	....	1.00
Iron, per cent	....	0.25
Phosphorus, per cent	....	0.05
Total other constituents, per cent	....	0.35

Revisions of Spec. for Copper-Silicon Alloy Rods, Bars, and Shapes (B 98 - 41)

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[16] Results of the letter ballot on this recommendation indicating its approval appear in the report of Committee B-5, see p. 186.

Revisions of Spec. for Copper-Silicon Alloy Wire for General Purposes (B 99 – 41)
Revisions of Spec. for Phosphor Bronze Sheet and Strip (B 103 – 40)

Withdrawal of Standard:

Spec. for Muntz Metal Condenser Tube Plates (B 57 – 27), replaced by Tentative Specifications for Copper-Alloy Condenser Tube Plates (B 171 – 42 T)

Committee B-1 on Copper and Copper-Alloy Wires for Electrical Conductors:

Report, not preprinted, presented from manuscript by J. H. Foote, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Rope-Lay-Stranded Copper Conductors Having Concentric-Stranded Members for Electrical Conductors
Spec. for Rope-Lay-Stranded Copper Conductors Having Bunch-Stranded Members for Electrical Conductors
Spec. for Bunch-Stranded Copper Conductors for Electrical Conductors

Adopted as Standard:

Spec. for Soft Rectangular and Square Copper Wire for Electrical Conductors (B 48 – 40 T), as revised

Withdrawal of Tentative Standard:

Spec. for Rope-Lay-Stranded and Bunch-Stranded Copper Cables for Electrical Conductors (B 158 – 41 T), replaced by the three new tentative specifications listed above.

Committee B-2 on Non-Ferrous Metals and Alloys:

Report presented, in the absence of the chairman, by Sam Tour, and the following actions taken:

Adopted as Standard:

Spec. for Phosphor Tin (B 51 – 38 T)
Revisions of Spec. for Lake Copper Wire, Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (B 4 – 27)
Revisions of Spec. for Electrolytic Copper Billets, Ingots, and Ingot Bars (B 5 – 27)

Committee E-2 on Spectrographic Analysis:

A progress report was presented from manuscript, in the absence of the chairman, by B. F. Scribner, secretary.

Committee E-3 on Chemical Analysis of Metals:

Report presented by G. E. F. Lundell, chairman, and the following actions taken:

Accepted as Tentative:

Chemical Analysis of Magnesium and Magnesium Alloys
Chemical Analysis of Aluminum and Aluminum Alloys, to replace Tentative Methods B 40 – 36 T
Chemical Analysis of Brasses, to replace Tentative Methods B 45 – 36 T
Chemical Analysis of Pig Lead, to replace Tentative Methods B 35 – 36 T
Chemical Analysis of Metallic Materials for Electrical Heating, to replace Tentative Methods B 71 – 34 T
Chemical Analysis of Nickel, to replace Tentative Methods B 41 – 36 T
Chemical Analysis of Slab Zinc (Spelter), to replace Tentative Methods B 38 – 36 T

Adopted as Standard:

Methods of Chemical Analysis for Aluminum, Columbium, and Lead in Steel (E 30 – 41 T), to be incorporated in Standard E 30 – 39.
Methods of Chemical Analysis of Ferro-Alloys (E 31 – 38 T)
Methods of Sampling Ferro-Alloys (E 32 – 39 T)

Paper:

The following paper was presented:

The Creep Characteristics of Some Copper Alloys at Elevated Temperatures—H. L. Burghoff, A. I. Blank, and S. E. Maddigan, presented by Mr. Burghoff.

FOURTEENTH SESSION—IRON

THURSDAY, JUNE 25, 8 P.M.

Session Chairman: President G. E. F. Lundell

Committee A-7 on Malleable Iron Castings:

Progress report presented from manuscript by F. L. Wolf, chairman.

Committee A-3 on Cast Iron:

Report presented by J. W. Bolton, chairman, and the following actions taken:

Accepted as Tentative:

Compression Testing of Cast Iron
Revision of Def. of Terms Relating to Cast Iron (A 196 – 36 T)

Adopted as Standard:

Spec. for Cast Iron Soil Pipe and Fittings (A 74 – 39 T)
Revision of Spec. for Gray-Iron Castings for Valves, Flanges, and Pipe Fittings (A 126 – 40)

Withdrawal of Standard and Tentative Standard:

Spec. for Cast-Iron Locomotive Cylinders (A 45 – 14)
Spec. for Chilled-Tread Cast-Iron Wheels (A 46 – 30 T)

It was announced that the committee would present to the Society through Committee E-10 on Standards, subsequent to the annual meeting, a proposed Tentative Recommended Practice for Torsion Testing of Cast Iron.[17]

Committee A-2 on Wrought Iron:

Report presented, in the absence of the chairman, by the chair, and the following actions taken:

Adopted as Standard:

Spec. for Single and Double Refined Wrought-Iron Bars (A 189 – 39 T)

Reaffirmation of Standards:

Spec. for Common Iron Bars (A 85 – 36)
Spec. for Refined Iron Bars (A 41 – 36)
Spec. for Riveted Steel and Wrought-Iron Pipe (A 138 – 34)
Def. of Terms Relating to Wrought Iron (A 81 – 33)

Sectional Committee A21 on Specifications for Cast-Iron Pipe and Fittings:

Informal progress report presented, in the absence of the chairman, by the chair.

Papers:

The following papers were presented:

Some Creep Studies on Cupola Malleable Cast Iron—J. J. Kanter and Glen Guarnieri, presented by Mr. Guarnieri.
Compression Testing of Cast Iron—W. Leighton Collins, presented by the author.
A Study of the Chemical, Physical and Mechanical Properties of Permanent Mold Gray Iron—Richard Schneidewind and Edward C. Hoenicke, presented by Mr. Schneidewind.
The Notch Sensitivity of Alloyed Cast Irons Subjected to Repeated and Static Loads—W. Leighton Collins and James O. Smith, presented by Mr. Collins.

FIFTEENTH SESSION—CEMENTITIOUS MATERIALS

THURSDAY, JUNE 25, 8 P.M.

Session Chairman: Frank E. Richart.

Committee C-1 on Cement:

Report presented by P. H. Bates, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Treated Portland Cement for Concrete Highway Pavements, this recommendation, not preprinted, was accepted by unanimous consent, subject to favorable letter ballot of Committee C-1[18]

Revisions of Chemical Analysis of Portland Cement (C 114 – 40 T):
- New alternate method for the rapid determination of sodium oxide and potassium oxide in cement
- New method of determining sulfide sulfur in cement
- New method for determination of sulfur trioxide in portland cement by means of the turbidimeter, this recommendation, not preprinted, was accepted by unanimous consent, subject to favorable letter ballot of Committee C-1[18]

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[17] See report of Committee A-3, Editorial Note, p. 110.

[18] Results of the letter ballot on this recommendation indicating its approval appear in the report of Committee C-1, see p. 231.

Adopted as Standard:

Test for Fineness of Portland Cement by Means of the Turbidimeter (C 115 – 41 T)

Revisions of Spec. for Portland Cement (C 150 – 41), comprising the following changes, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee C-1:[18]

Section 3.—Change the definition of portland cement to read as follows by the addition of the italicized words and the omission of those in brackets:

> Portland cement is the product obtained by pulverizing clinker consisting essentially of hydraulic calcium silicates, to which no additions have been made subsequent to calcination other than water and/or untreated calcium sulfate, except that *additions not to exceed 1.0 per cent of* other materials may be *interground with the clinker at the option* of the manufacturer [added] provided that *such* [other] materials *in the amounts indicated have been shown to be not harmful* [may be added provided that they have been shown to be acceptable, in the amounts indicated,] by tests carried out or reviewed by Committee C-1.

In the note and footnote accompanying the definition, substitute the phrase "not harmful" for the word "accepted" wherever it appears.

Table II.—Change the third sentence in footnote *c* from its present form: namely,

> Tests at 28 days on types I and II cement may be waived at the option of the purchaser.

to read:

> Unless otherwise specified, the compressive and tensile strength tests on types I and II cement will be made only at 3 and 7 days.

Revisions of Methods of Chemical Analysis of Portland Cement (C 114 – 40), with the following changes, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee C-1:[18]

Section 3.—Change the requirements for balances to read as follows:

> 3. (*a*) *Balances.*—The analytical balances used in the chemical determinations shall conform to the following requirements:
>
> Capacity not less than 100 g. in each pan,
>
> The two arms of the beam equal within (*1*) two parts in 100,000 for balances in use, and (*2*) one part in 100,000 for new balances (Note 1),
>
> Capable of reproducing results within 0.0002 g,
>
> Sensibility reciprocal[a] not more than 0.0005 g. per division of the graduated scale for any load up to 100 g. (Note 2). In case of direct-reading balances this requirement does not apply, and
>
> Any rapid weighing device that may be provided, such as a chain, damped motion, or heavy riders, shall not introduce errors greater than 0.0001 g. at any reading and with any allowable load on the scale pans.
>
> NOTE 1.—The lengths and ratios of the arms may change with use, and with age even if not used.
>
> NOTE 2.—If an auxiliary metal or glass pan is used, its weight is a part of the load.
>
> (*b*) Weights.—The weights used in the chemical determinations shall conform to the requirements of the National Bureau of Standards specifications for class "S" weights as contained in Bureau of Standards *Circular No. 3*, "Design and Test of Standards of Mass." The weights (including the riders) shall be checked when questioned or at least once a year with weights that have been certified by the National Bureau of Standards within 5 yr. They shall be adjusted within the tolerances given in the above-mentioned specifications for class "S" weights, according to their apparent mass as determined by comparison with brass standards in air.

Section 5.—Change the first sentence from its present form to read as follows:

> Blank determinations shall be made on approximately the same amount of reagents and using the procedures described in Sections 6, 7, 11, 13, 15, 17, 18, 21, 22, 24, 28, 30, and 31.

Section 31.—Change the test procedure[b] for insoluble residue to read as follows:

> 31. To 1 g. of the sample, add 10 ml. of cold water and 5 ml. of HCl (sp. gr. 1.19). Warm the solution gently and grind the material with the flattened end of a glass rod for a few minutes until it is evident that decomposition of the cement is complete (Note). Dilute the solution to 50 ml. and digest for 15 min. at a temperature just short of boiling. Filter the residue, wash six times with hot water, and digest the filter paper and contents in 100 ml. of NaOH solution (1 per cent), the solution being held at a temperature just short of boiling for 15 min. Using methyl red as indicator, acidify the solution with HCl and add an excess equivalent to 4 or 5 drops of HCl (sp. gr. 1.19). Filter and wash the residue 12 to 15 times with hot NH_4Cl solution (2 per cent). Ignite the residue in a tared crucible at 900 to 1000 C., cool it in a desiccator and report the gain in weight as insoluble residue.
>
> NOTE.—If a sample of portland cement contains an appreciable amount of manganic oxide, there may be brown compounds of manganese which dissolve slowly in cold diluted HCl but rapidly in hot HCl in the specified strength. In all cases dilute the solution as soon as decomposition is complete.

Committee C-12 on Mortars for Unit Masonry:

Report presented by J. W. McBurney, chairman, and the following action taken:

Accepted as Tentative:

Revision of Specs. for Aggregate for Masonry Mortar (C 144 – 39 T)

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[a] The sensibility reciprocal is a measure of the sensitiveness of a balance and is the weight required to move the position of equilibrium of the pointer one division. For a complete definition of sensibility reciprocal, see National Bureau of Standards *Handbook H22*, "Specifications, Tolerances, and Regulations for Commercial Weights and Measures and Weighing and Measuring Devices," pp. 68–69.

[b] This revised method was proposed by California Portland Cement Co., where it had been specially developed for use with blended cements. This method or any other method designed for the estimation of acid-insoluble substance in any type of cement is empirical, because the amount obtained depends on the reagents and the time and temperature of digestion. If the amount is large, there may be a little variation in duplicate determinations. The directions should be followed closely in order to reduce the variation to a minimum. When the method is used on blended cement, the decomposition in acid is considered to be complete when the portland cement clinker is decomposed completely. A NH_4Cl solution is used in the final washing to prevent finely ground insoluble material from passing through the filter paper.

It was announced that the proposed Tentative Specifications for Mortar for Concrete Unit Masonry which appeared in the report as preprinted failed to receive the necessary two-thirds vote in the committee, and these specifications were accordingly not presented to the Society.

Committee C-7 on Lime:

Report presented by N. C. Rockwood, chairman, and the following actions taken:

Adopted as Standard:

Spec. for Hydraulic Hydrated Lime for Structural Purposes (C 141 – 38 T)

Adopted as Standard:

Revision of Spec. for Quicklime and Hydrated Lime for Silica Brick Manufacture (C 49 – 24)

Committee C-11 on Gypsum:

Report presented, in the absence of the chairman, by B. E. Kester, and the following actions taken:

Accepted as Tentative:

Revisions of Spec. for Calcined Gypsum for Dental Plasters (C 72 – 40)

Revisions of Tests for Gypsum and Gypsum Products (C 26 – 40)

Adopted as Standard:

Revisions of Spec. for Gypsum Lath (C 37 – 30)

Revisions of Spec. for Gypsum Wall Board (C 36 – 34)

Revisions of Spec. for Gypsum Sheathing Board (C 79 – 34)

Revisions of Tests for Gypsum and Gypsum Products (C 26 – 40)

Withdrawal of Tentative Revision of Standard:

Spec. for Gypsum (C 22 – 41)

The recommendation for adoption as standard of the revised definition for "perforated gypsum lath" was discussed on behalf of Committee E-8 on Nomenclature and Definitions. In consideration of this definition in Committee E-8 the question was raised whether it is a definition or specification. Attention was called to the fact that the first sentence would appear to define the material, but that the remainder is in the nature of a specification. Committee E-8 requested that this recommendation be deferred for a year in order that the proposed revised definition might be discussed with Committee C-11. The recommendation for adoption failed of approval, and was referred back to the committee.

Papers:

The following papers were presented:

Long-Time Volume Changes of Portland Cement Bars—Alfred H. White and Harold S. Kemp, presented by Mr. White.

Theory and Measurement of Plasticity and Workability of Mortars—Paul S. Roller, presented by the author.

Curing of Masonry Mortars—Howard R. Staley, presented by the author.

The Effect of Brick Absorption Characteristics upon Mortar Properties—F. O. Anderegg presented by the author.

SIXTEENTH SESSION—BUILDING MATERIALS, CONCRETE AND CONCRETE AGGREGATES

FRIDAY, JUNE 26, 9.30 A.M.

Session Co-Chairmen: Stanton Walker; T. A. Fitch

Committee C-18 on Natural Building Stones:

Report, in absence of chairman, presented by title.

Committee C-15 on Manufactured Masonry Units:

Report presented by D. E. Parsons, chairman, and the following actions taken:

Accepted as Tentative:

Revision of Methods of Sampling and Testing Brick (C 67 – 41)

Adopted as Standard:

Spec. for Sewer Brick (Made from Clay or Shale) (C 32 – 39 T)

Spec. for Paving Brick (C 7 – 40 T)

The paper by John W. McBurney on "Relations Between Results of Laboratory Freezing and Thawing and Several Physical Properties of Certain Soft-Mud Bricks," to be appended to the report, was presented by the author.

Committee C-8 on Refractories:

Report presented, in the absence of the chairman, by G. Bickley Remmey, and the following actions taken:

Accepted as Tentative:

Spec. for Fireclay Plastic Refractories for Boiler Furnaces and Incinerator Service

Adopted as Standard:

Def. of Terms Relating to Refractories (Abrasion, Burning, Corrosion, Erosion, and Slagging) (C 71 – 40 T)

Revision of Testing Insulating Fire Brick (Compressive Strength, Flexural Strength, and Permanent Linear Change After Heating) (C 93 – 41)

Revisions of Test for Pyrometric Cone Equivalent of Refractory Materials (C 24 – 35)

Revisions of Panel Test for Resistance to Thermal and Structural Spalling of Refractory Brick (C 38 – 36)

Revisions of Panel Test for Resistance to Thermal and Structural Spalling of High Heat Duty Fireclay Brick (C 107 – 40)

Revisions of Panel Test for Resistance to Thermal and Structural Spalling of Super Duty Fireclay Brick (C 122 – 40)

Committee C-16 on Thermal Insulating Materials:

Report presented, in the absence of the chairman, by E. T. Cope, secretary, and the following action taken:

Accepted as Tentative:

Test for Thermal Conductivity of Materials by Means of the Guarded Hot Plate

The paper by F. C. Houghten on "Conductivity Determination by the Guarded Hot-Plate Method," appended to the report, was presented by the author.

Committee C-9 on Concrete and Concrete Aggregates:

Report presented by F. H. Jackson, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Waterproof Paper for Curing Concrete

Sampling Fresh Concrete

Measuring Length of Drilled Concrete Cores

Test for Air Content of Freshly Mixed Concrete

Revisions of Def. of Terms Relating to Concrete and Concrete Aggregates (C 125 – 36 T)

Revisions of Spec. for Ready Mixed Concrete (C 94 – 41 T), with revisions in the report as preprinted, except that the revised specifications were accepted by unanimous consent, subject to favorable letter ballot of Committee C-9,[19] for publication as tentative rather than for adoption as standard as recommended in the report as preprinted.

Adopted as Standard:

Revisions of Spec. for Concrete Aggregates (C 33 – 40), with the immediate adoption of a revision in Section 3 (*a*), also adoption of the tentative revision published since 1941 rather than publication as tentative, modified in accordance with the report as preprinted. This recommendation was accepted by unanimous consent, subject to favorable letter ballot of Committee C-9.[19]

Revisions of Method of Making and Storing Compression Test Specimens of Concrete in the Field (C 31 – 39), with the tentative revision published since August, 1941, recommended for adoption as standard, and with the following further revision in place of the editorial change given in the report as preprinted, this recommendation was accepted by unanimous consent, subject to favorable letter ballot of Committee C-9:[19]

Section 5.—Change from its present form, to read as follows:

> 5. Samples of concrete for test specimens shall be taken in accordance with the Tentative Method of Sampling Fresh Concrete (A.S.T.M. Designation: C 172) of the American Society for Testing Materials. The location in the work of the batch of concrete thus sampled shall be noted for further reference.

Revisions of Test for Cement Content of Hardened Portland-Cement Concrete (C 85 – 36)

Revisions of Test for Unit Weight of Aggregate (C 29 – 39)

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[19] Results of the letter ballot on this recommendation indicating its approval appear in the report of Committee C-9, see p. 246.

Revisions of Test for Compressive Strength of Concrete (C 39 – 39), with the tentative revision published since June, 1940, and also the tentative revision submitted in August, 1941, accepted by unanimous consent for adoption as standard, subject to favorable letter ballot of Committee C-9.[19]

Revisions of Spec. for Lightweight Aggregates for Concrete (C 130 – 39)

Revisions of Method of Securing Specimens of Hardened Concrete from the Structure (C 42 – 39)

Revisions of Test for Structural Strength of Fine Aggregate Using Constant Water-Cement-Ratio Mortar (C 87 – 39)

Revisions of Test for Compressive Strength of Concrete Using Portions of Beams Broken in Flexure (C 116 – 39)

Revisions of Test for Specific Gravity and Absorption of Coarse Aggregate (C 127 – 39)

Revisions of Test for Specific Gravity and Absorption of Fine Aggregate (C 128 – 39)

This recommendation, not preprinted, for the adoption of pending tentative revisions in the above six standards, was accepted by unanimous consent, subject to favorable letter ballot of Committee C-9.[19]

Withdrawal of Standards:

Test for Voids in Inundated Fine Aggregate (C 69 – 30)

Spec. for Ready Mixed Concrete (C 94 – 38), this recommendation, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee C-9.[19]

Editorial Changes Accepted:

Test for Compressive Strength of Concrete (C 39 – 39)

Sanford E. Thompson Award:

Mr. Jackson introduced Mr. Sanford E. Thompson who presented the Sanford E. Thompson Award. The award was established by Committee C-9 in honor of the first chairman of the committee, and for the purpose of stimulating research and encouraging investigations that extend the knowledge of concrete and concrete aggregates.

Mr. Thompson then announced that the Sanford E. Thompson Award had been made to Mr. John Tucker, Jr. Chief, Concreting Materials Section, National Bureau of Standards, for his paper on "Statistical Theory of the Effect of Dimensions and of Method of Loading upon the Modulus of Rupture of Beams," presented at the 1940 annual meeting. In the absence of the recipient, D. E. Parsons accepted from Mr. Thompson the certificate of the Sanford E. Thompson Award, expressing appreciation on behalf of Mr. Tucker for the honor conferred.

Papers:

The following papers were presented:

The Influence of the Durability of Aggregate upon the Durability of the Resulting Concrete—H. H. Munger, abstract presented by the chair.

Effect of Height of Test Specimens on Compressive Strength of Concrete[20]—James W. Johnson, presented by the author.

Factors Influencing the Reflectivity of White Portland Cement Concretes—C. W. Muhlenbruch and E. H. Miller, presented from manuscript by Mr. Muhlenbruch.

Powdered Aluminum as an Admixture to Concrete—Roy W. Carlson, presented from manuscript by the author.

There being no further business the chairman then declared the Forty-fifth Annual Meeting adjourned *sine die.*

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[20] Published in the ASTM BULLETIN, No. 120, January, 1943, p. 19.

CHEMICAL REQUIREMENTS AND CHEMICAL ANALYSIS

ANNUAL ADDRESS BY THE PRESIDENT

G. E. F. LUNDELL[1]

JUNE 24, 1942

A few remarks may be in order before I present my prepared address: This has been a year of opportunity and responsibility for our Society. We have not been unprepared, for our efforts in connection with the national preparedness program have stood us in good stead in the far more serious business of the war that has come upon us. For months prior to "December 7" our committees, in close cooperation with the military branches, had been intensifying their specifications work to cover the rapidly changing situation in the materials field. Our Society had stream-lined the procedures under which its standards are set up. As a result, some 56 Emergency Standards and Emergency Alternate Provisions in Standards have already been drawn up in the interest of conserving scarce materials and to expedite the production of materials.

We have had the opportunity to demonstrate the value of groups trained in research on materials, in the standardization of methods of testing, and in formulating standard specifications. We are proud of the roster of the members of our Society who are officially connected with the country's war effort. I wish to express the Society's thanks to its members for their loyal and unselfish cooperation and splendid effort in a year fraught with grave problems. When victory is won and we have had time to take stock, I have no doubt whatever that the Society will be proud of its war record.

Presidential addresses in the past have dealt with many phases of our Society's activities, but none, so far as I have been able to determine, has dealt with chemical analysis. This will be my excuse for here recording sundry observations based on a quarter of a century's contact with chemical requirements and with chemical analysis in the A.S.T.M. What I have to say is not by way of criticism of our chemical requirements or of apology for our analysts, but rather with the desire that the problems encountered by the analyst may be better appreciated, and that these may be kept in mind when chemical requirements are set up, or the analyst's work is appraised.

Analyses to check chemical requirements are a most important phase of the testing of materials covered by the specifications of our Society. One would expect, therefore, that most of our members would be entirely familiar with chemical analysis and its problems. Quite the reverse is true, for many have but a hazy notion as to what it is all about. The reasons for this are not difficult to establish, even though one must start from the beginning.

In the earliest days, analyses were made by those who had need of them, and the roster of analysts included the names of some of our most illustrious chemists. In those days each user of the analysis was a proper judge of the accuracy that was possible or needed, of the labor required, of the time that must be spent on the analysis, and of all of the other considerations that might be involved. Analyses were made leisurely, their costs were not questioned, and complete analyses were the rule, not the exception.

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[1] Chief, Chemistry Division, National Bureau of Standards, Washington, D. C.

The old order of analysis has to all intents and purposes died out. Today, very few analyses are made by those who need them, and many who need analyses have but little knowledge of the difficulties that may be involved, the labor that must be expended, or the results that can rightfully be expected.

In the industrial field, a contributing factor to the present-day misconception of chemical analysis can be traced to the courses in so-called chemical analysis which must be taken by engineering students in many of our colleges and universities. Many of these students later employ analysts. The courses necessarily deal with chemical determinations that are so simple that they do not deserve the label "chemical analysis" at all, and leave the student with an entirely erroneous conception of what chemical analysis, particularly applied chemical analysis, is like.

The steel-works' analyst doesn't find his sulfur in the form of a pure water-soluble sulfate. He finds it combined with iron as the sulfide and associated with a number of elements, and he must not only convert the sulfide to soluble sulfate, but also provide for the compounds that interfere, before the academic procedure can be applied.[2] In other words he now deals with chemical analysis as well as with a determination. The operations of chemical analysis may be divided into those that make the determination possible, and those that attend the determination itself. What is not generally understood is that in applied analysis the former are often by far the most important. The relation of the analysis to the determination might be compared with that between the art of fishing and the measurement of the fish. The catching of the fish may be quite difficult and require considerable planning and patience. On the other hand, almost anyone can measure or weigh the fish after it has been caught.

In the schools, a contributing factor in the divorce of chemical determinations from chemical analysis was a swing from the applied toward the theoretical aspects of analytical chemistry which took place toward the close of the last century. In this, the cart was put before the horse, the determination was elected to pull the analysis instead of the analysis to pull the determination. This left such illustrious analysts as Hillebrand in the category of "cook-book" chemists, and attempted to substitute a strange hybrid disporting in a field of his own making, a field that was chiefly physical chemistry, and that ignored inorganic chemistry, which is the proving ground of analytical chemistry. The chemist who was working in the field of applied analysis was told that he was using primitive methods and uncalibrated tools, and was given a map of a promised land which was free from the weeds of ignorance, and ripe for tilling. Some have never come back to the old homestead, some have returned with a sigh of relief, and all agree that it was an "experience."

No one will deny that it is important to know the best way to proceed when one reaches the determination point, but it must not be forgotten that in applied analysis there are often many detours, and that it may be very difficult to reach that point.

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[2] Throughout the discussion it is assumed that the analyst has obtained a representative and properly prepared sample for analysis. This is one of the analysts' important problems; sometimes it presents greater difficulties than the analysis. An adequate treatment of sampling would require more time than can be spared here.

Twenty-five years ago in a lecture on "Our Analytical Chemistry and Its Future," Hillebrand[3] referred to a talk along similar lines given some 12 years[4] earlier in which he had expressed his conviction that our educational institutions must bear a large share of the blame for the unsatisfactory condition in which the art of analysis had fallen. In his lecture he then asked "Have they improved?" and answered "Candor compels me to say that evidences of improvement are few." There is still room for improvement. It would be helpful to the understanding of analytical chemistry if the term "chemical analysis" were reserved for true analysis, and if most of our courses in so-called analytical chemistry were labeled elementary or advanced chemical determinations.

STANDARD METHODS OF ANALYSIS

The greatest handicap under which the analyst works is that the chemical methods which he uses are neither truly specific nor applicable in all environments. He has made but little progress, and perhaps never will, in reaching the Shangri La of his analytical dreams, a land in which he has a shelf of 92 specific reagents, one for each of the elements. As a consequence, a thoroughly explored and most accurate method for the determination of a component in a given material may yield grossly inaccurate results if it is used for the determination of the same component under slightly changed conditions. For example, present-day electrolytic methods for the determination of copper in brasses or steels cannot be used if these alloys contain silver, intentionally added or introduced through scrap. To meet such a development, a new method must be drawn up immediately, in which provision is made for the removal of the silver before the electrolysis is started, or for its presence in the deposit that is obtained. The latter of course would necessitate a supplementary determination of silver, either in the deposit or in the material itself. Similar considerations hold whenever the analyst enters any new field with an established method or plans to apply a new method under conditions that are different from those under which it has been tested.

In the early days each analyst selected his own methods or devised them. Usually there was no opportunity to make proper tests of the accuracy of the methods as applied to the materials in hand, with the result that quite often shaky or inappropriate methods were used, results were misleading, and misunderstandings and disputes were common. With the advent of specifications and chemical requirements two important developments were inevitable: the formulation of standard methods, and the preparation and distribution of standard analyzed samples.

Standard methods of analysis, except those of the pharmacy, had their beginnings with chemists interested in the fertilizer industry in this country in the early eighties. In the late eighties and early nineties, the state of iron and steel analysis, here and abroad, called for similar action, and an American subcommittee, under the lead of one of the incorporators of our Society, Dr. Dudley, made an elaborate study of methods of steel analysis. These subsequently entered in large measure into the early standards adopted by the A.S.T.M. It seems strange at this date that a considerable part of this work had to do with a study of the now discarded method for the determination of carbon in irons or steels by attack with neutral or acid solutions of the double chloride of copper and ammonium or of copper and potassium, followed by ignition of the carbonaceous residue in oxygen, or its oxidation in chromic and sulfuric acids.

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[3] Chandler Foundation Lecture delivered at Columbia University, November 27, 1916; *Journal of Industrial and Engineering Chemistry*, Vol. 9, p. 170 (1917).

[4] *Journal*, Am. Chemical Soc., Vol. 27, p. 300 (1905).

Since that time a great deal of effort has been spent in our country not only in formulating standard methods as needed, but also in revising existing ones, for these are ever changing to make way for better methods, to meet changes in the composition of the materials, or to satisfy demands for greater accuracy or speed.

The amount of time and effort that may be spent in formulating standard methods can be illustrated by the work which has been done by A.S.T.M. Committee E-3 on Chemical Analysis of Metals in developing the tentative standard methods of chemical analysis of admiralty metal and of free-cutting yellow brass which are being recommended for adoption at this annual meeting. These are the result of thorough discussions of possible methods in meetings and in correspondence, final agreement on methods for six components of the admiralty metal and five components of the brass, the preparation of suitable samples for test, and the testing of the methods against the samples in hundreds of analyses by the committee members. The results are shown in Table I.

TABLE I.—RESULTS OBTAINED IN TESTS OF PROPOSED TENTATIVE A.S.T.M. METHODS.

Constituent	Spread of Results, per cent	Number of Results		Mean, per cent[a]	Average Deviation from Mean, per cent[a]
		Reported	Rejected		
ADMIRALTY METAL					
Copper	69.90 to 70.04	33	1	69.99	0.02
Zinc	28.62 to 29.05	20	7	28.90	0.02
Tin	0.971 to 1.03	28	3	0.998	0.010
Antimony	0.040 to 0.056	24	1	0.050	0.003
Arsenic	0.031 to 0.063	26	2	0.057	0.004
Phosphorus	0.038 to 0.050	19	1	0.047	0.001
FREE-CUTTING YELLOW BRASS					
Copper	62.14 to 62.28	43	0	62.21	0.03
Zinc	34.11 to 34.90	20	7	34.49	0.04
Lead	2.92 to 3.06	40	1	3.00	0.03
Nickel	0.19 to 0.21	23	0	0.20	0.004
Iron	0.099 to 0.115	23	1	0.105	0.002

[a] Excluding results rejected according to Chauvenet's criterion.

In the lecture which I have already cited, Dr. Hillebrand stated that "My experience makes me feel strongly that the advances in chemical analysis to be expected through committees of professional societies are too often slight and are secured at a cost of time and effort quite incommensurate with the gains." This was one of the rare occasions when the doctor was wrong in an analysis, for great contributions toward the betterment of methods of chemical analysis have been made by technical committees during the past 25 years. This is particularly true in the A.S.T.M. through Committee E-3 on Chemical Analysis of Metals, the Working Committee on Methods of Chemical Analysis of Cement of Committee C-1 on Cement, Section E on Analysis of Committee C-8 on Refractories, Subcommittee II on Chemical Analysis of Committee C-14 on Glass and Glass Products, Committee E-2 on Spectrographic Analysis, and the numerous subcommittees on chemical analysis in the fields covered by the D committees.

As a result of such efforts, there have been developed by interested groups a large number of standard methods of chemical analysis to cover their needs. Among these are methods, such as those of the United States Pharmacopoeia, of the Association of Official Agricultural Chemists, of the United States Treasury Dept., and of those contained in Federal Specifications for Portland Cement, which are recognized by courts of law in the United States; methods not having legal recognition but approved by scientific or technical organizations such as the A.S.T.M. and the American Chemical Society; and last but not least the thousands of methods that have been developed by industries and utilities to meet their daily analytical problems. Many of the company methods are drawn up with at least as much care as are the official methods, and most of them are periodically tested for performance against company standards and, if need be, for the conformance of results with those obtained by standard methods with which they may be compared.

As for conditions in countries other than our own, a report of the Standard Methods of Analysis Subcommittee of the Committee on the Heterogeneity of Steel Ingots,[5] just released by the Iron and Steel Institute of Great Britain, carries complaints and suggested remedies that have a familiar ring: "Strong views were expressed regarding the accuracy of existing methods of analysis, upon the results of which casts of steels were liable to rejection, and it was decided that a subcommittee of qualified metallurgical chemists should be asked to review existing methods and devise standard reliable processes."

A criticism common to most standard methods of analysis is that no tolerances are given to guide the judgment of the user of the method or of the recipient of the analysis. Both should know whether differences such as 0.1 per cent between the results for silica reported by two chemists working on the same sample of a silicate are normal or abnormal. A start in giving such information is now going forward, as in the Tentative Methods of Chemical Analysis of Soda-Lime Glass (C 169 - 41 T)[6] which indicate the probable accuracy of the results that are obtained, and in the Standard Methods of Chemical Analysis of Refractory Materials (C 18 - 41)[7] which indicate the permissible variations between check determinations made as described in the methods. A discouraging feature in compiling such data is that after they have been gathered the findings are applicable in tests made on the specified material, and do not necessarily apply if the composition of the material is changed by the introduction of new constituents or even by disturbing the relative amounts of the constituents.

STANDARD SAMPLES

Proposals concerning the preparation and distribution of standard samples for use in checking methods of chemical analysis also had their beginnings in the eighties. In a paper "On International Standards for the Analysis of Iron and Steel" presented in 1889 before the Engineer's Society of Western Pennsylvania, John W. Langley observed "A practical remedy for most of these differences of analytical reports can be found in a system of international standards of iron or steel, whose composition shall be most rigidly and authoritatively determined by committees of chemists and metallurgists, appointed in the most important iron-producing countries. These committees propose to analyze a set of samples which shall be prepared in one place and distributed to the several countries; after the committees have made their reports, the standards thus established will probably be deposited in some public place, and small portions of them can then be issued to properly-qualified applicants. A chemist obtaining a sample of the standards can then check his own work by them, and thus he can determine a factor by which in future his results must be multiplied to make them agree with the standards. It is confidently believed that the plan, if successfully carried out, will result in a very considerable degree of uniformity in the analysis of these important metals in different countries and will greatly lessen those scientific and commercial disputes which now sometimes assume formidable proportions." The elusive factors by which analysts' results must be multiplied to make them agree with the standards were never determined, for after five samples were prepared and considerable work was done, the project fell through, and the samples were never distributed as planned.

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[5] A joint committee of the Iron and Steel Institute and the British Iron and Steel Federation, reporting to the Iron and Steel Industrial Research Council.

[6] 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 300.

[7] *Ibid.*, p. 67.

In the meantime, the American Foundrymen's Assn. prepared and distributed standardized pig-iron samples, comprising four sets and covering a considerable range of compositions. In 1905 these samples were turned over to the National Bureau of Standards. The announcement by Dr. Moldenke, then secretary of the American Foundrymen's Assn., and for a number of years the chairman of our Committee A-3 on Cast Iron is interesting: "We beg to announce the taking over by the Government of our Standardizing Bureau, and that hereafter the Standardized Drillings of Cast Iron prepared and sold by our Association are to be obtained from the Bureau of Standards of the Department of Commerce and Labor. The American Foundrymen's Assn. has been highly honored by this recognition of its work, and the undoubted broadening out of the preparation of standard drillings now to be expected will greatly enhance the value of standards for check determinations in the iron and steel industry.

"It is but fitting to recall that to our President, Mr. Thomas D. West, belongs the credit of first calling attention to the advantages such a series of cast iron borings, duly standardized by the ablest chemists of our country, would be to iron laboratories. To him for the successful building up of the enterprise, as well as to Professor Smith of the Case School of Applied Science, for the details of manipulation, chemists all over the world are grateful, for the standards of the Association are accepted everywhere as arbiters in case of dispute."

In the same year, committees of the American Chemical Society on Uniformity of Technical Analysis and on Zinc Ore Analysis assigned to the Bureau their samples of argillaceous limestone and zinc ore. From this start, the work has grown so that now it covers some 130 samples. Most of these are certified as to their chemical composition or performance; the remainder are certified as to certain physical properties, or are distributed as standards of reference.

Samples certified as to chemical composition or performance include ores, refractories; ceramic materials, irons, plain carbon and alloy steels, steel-making alloys, non-ferrous metals such as brasses, bronzes, bearing metals, solders, zinc- and aluminum-base alloys, and pure chemicals. Samples certified for physical properties include materials certified for their fineness, for their melting points, and for thermoelectric, colorimetric, and saccharimetric values. Standards of reference, which have been compared with master samples at the Bureau or measured for compliance with an arbitrary standard include such diverse materials as radon and gamma-ray standards, glass standards for the calibration of spectrophotometers, Lovibond glasses for colors of oils, etc., glass opacity standards for the paper industry, standard oils for viscosity, standards of radiation and lamp standards of color temperature, and standard finish samples for builders' hardware, and colors for sanitary ware, kitchen and bathroom accessories. A start has also been made to cover special analytical fields such as pH, microanalytical and spectrographic.

Theoretically, it would be desirable to prepare standard samples covering all of the materials that are analyzed by the chemist. In such a case, it would be necessary to prepare samples covering the different environments in which an element may be found, as well as the range of concentrations that may be encountered. Of the astronomical number of standards that would be needed in such a program, only an infinitesimal number can be prepared. In choosing the materials that are now offered, the guiding considerations have been the importance of the material in applied chemical analysis, and its stability when prepared for use as a standard. To this might be added the willingness of industry to aid in the work, for Government appropriations have never been generous. The growth of the standard sample work has therefore depended in no small degree on cooperation by industry, both in donating material and in giving generous aid in the analyses incidental to the certification of the samples.

It would be hard to overstate the very important part that the National Bureau of Standards Standard Analyzed Samples have played in the development of methods of chemical analysis and in improving their accuracy since the work was begun thirty-seven years ago. This has come about, first, through the very extensive cooperative work and correspondence incidental to the certification of the chemical compositions of the samples; second, through the use of the certified standards in checking methods of chemical analysis that are in daily use in science and industry; and, third, through the use of the samples in determining the reliability and accuracy of new methods of analysis.

Primary standard samples patterned along the lines of the N.B.S. standards have been prepared in other countries, notably in England and in the Soviet Union.

In addition to these primary standards, there must also be mentioned the thousands of secondary standards which have been prepared and standardized, often against the Bureau's samples, by analysts having need of large amounts of standardized material for constant checking of their work. To these secondary standards must be added others of materials not yet supplied by the N.B.S., which have been prepared and standardized along Bureau lines by analysts having need of them. Quite often the Bureau cooperates in their analysis.

If standard samples are available, there is a growing tendency to allow the analyst to use any method which yields acceptable results when applied to a standard sample of the material under test. The analyst is thereby given a double-barreled weapon which enables him to test the accuracy as well as the precision of his analytical aim.

PRECISION AND ACCURACY

We come now to considerations of the precision and the accuracy of the analyst's work. These terms are often used interchangeably and therefore incorrectly, for precision is a measure of reproducibility, while accuracy is a measure of correctness. The analyst is vitally interest in both, for his results must be sufficiently accurate for the purpose in mind, and he cannot achieve accuracy without precision, especially since his reported result is usually based on one determination and rarely on more than three determinations. The recipient of the analysis is interested in accuracy alone, and only in accuracy sufficient for his purposes. This may range all the way from 1 part in 10 as in the chemical requirements for phosphorus or sulfur in some steels, to 1 part in 10,000 as in the chemical requirements for some of our coppers. If low accuracy suffices, special efforts on the part of the analyst to achieve high accuracy are wasted.

It is a simple matter to determine the precision that can be expected if a method of chemical analysis is applied to a given material by a single analyst or by a group of qualified analysts, for all that is necessary is to gather the results, and treat them by suitable statistical methods. If the results alone are considered, all that can then be claimed for the mean and the indicated precision is that they represent what can be expected if the method is applied to the material in question. If the method is a proper one for the material the mean may well be close to the true value, and the indicated precision can be taken as a gage of the accuracy to be expected. If the method is unsuited to the material, identical results can only represent high precision, wasted effort, and misinformation, unless the analyst has been able to determine Dr. Langley's factor by which the results can be multiplied to insure their accuracy. In other words, if we pass from precision to accuracy, we must consider the *material* and the *method* as well as the results.

When an analyst uses a standard method which has been drawn up for the analysis of the material under test he is primarily interested in the precision of the results that are obtained, and parks his worries as to the accuracy of the results with the sponsor of the standard method. If he uses a standard sample of the material, he obtains a direct measure of the accuracy of his work. The analyst has therefore little excuse today for reporting grossly inaccurate work so long as he can stay in the fields covered by the standard methods or standard samples. If he enters unexplored fields, his troubles multiply in proportion to the degree of accuracy required, for operations designed to fix the accuracy are usually too costly of time, effort, and money to be carried by an isolated analysis. What these may entail can be illustrated by the determination of iron in the National Bureau of Standards standard Sibley iron ore, which involved the use of seven different methods of analysis, studies of their accuracy, determinations of ore constituents other than iron, calculations of their effects in the methods, and the selection of the best value for iron as indicated by the data.

From what has been said it will be evident that one cannot arrive at the most accurate value through inspection of results alone, whether they be a dozen results reported by a single analyst or by a dozen analysts. To go beyond the arithmetical mean one must have a reasonably detailed description of the methods used, and the analytical knowledge required to sift the good methods from the bad. From the viewpoint of the conscientious analyst this constitutes one of the most discouraging features of chemical analysis, for excellent work may yield a result so far out of line with that obtained in mediocre work that it might actually be viewed with suspicion by a reviewer who considers results alone.

A criticism that applies to most analysts is their failure to appraise the limitations of their methods of analysis, and to round off reported results accordingly. Reports for silica in silicate materials are still reported to the second decimal place in spite of the fact that in ordinary analyses it is difficult enough to get concordance in the first decimal place. Results that are properly rounded off should not be regarded as a confession of weakness on the part of the analyst, but rather as a warning that if greater accuracy is to be certified, special, and usually more costly and time-consuming, procedures must be used.

In their abuse of significant figures

there is little to choose between the metallurgist in drawing up chemical requirements in a specification and the analyst in reporting the results of his tests That the unbridled metallurgist is apt to go out of bounds may be illustrated by certain of our specifications. For example in the Standard Specifications for Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (B 5 - 27)[8] it is specified that the copper in all shapes shall have a purity of at least 99.900 per cent. Standard Specifications for Fire-Refined Copper Other Than Lake (B 72 - 33)[9] goes this one cipher better and specifies that the minimum content of copper shall be 99.7000 per cent! Legally speaking 99.69994 per cent copper would not pass. Analytically speaking, the analyst does well in obtaining an accuracy of 1 part

TABLE II.—CAR WHEEL IRON.
First Results Reported by 17 Analysts.

Constitutent	Spread of Results, per cent	Mean, per cent	Probable Value, per cent	Average Deviation from Probable Value, per cent
Carbon	3.03 to 3.12^a	3.07	3.06	0.02
Silicon	0.54 to 0.602^b	0.569	0.585	0.02
Sulfur (gravimetric)	0.122 to 0.144^c	0.133	0.134	0.005

[a] Excluding 2 results of 2.83 and 3.16.
[b] Excluding 1 result of 0.485.
[c] Excluding 1 result of 0.154.

TABLE III.—FERROMANGANESE.
First Results Reported by 13 Analysts.

Constituent	Spread of Results, per cent	Mean, per cent	Probable Value, per cent	Average Deviation from Probable Value, per cent
Manganese	79.80 to 80.35	80.09	80.07	0.15
Carbon	6.78 to 6.90^a	6.84	6.83	0.03
Silicon	0.766 to 0.843	0.797	0.810	0.02

[a] Excluding 2 results of 6.55 and 6.70.

TABLE IV.—MOLYBDENUM-TUNGSTEN STEEL.
First Results Reported by 11 Analysts.

Constituent	Spread of Results, per cent	Mean, per cent	Probable Value, per cent	Average Deviation from Probable Value, per cent
Molybdenum	6.75 to 7.21	7.03	7.07	0.10
Tungsten	5.98 to 6.34	6.20	6.29	0.11
Manganese	0.216 to 0.272	0.252	0.250	0.01
Phosphorus	0.020 to 0.031	0.026	0.027	0.003

TABLE V.—BRASS.
First Results Reported by 9 Analysts.

Constituent	Spread of Results, per cent	Mean, per cent	Probable Value, per cent	Average Deviation from Probable Value, per cent
Copper	69.98 to 70.18	70.07	70.07	0.04
Zinc	27.12 to 27.32^a	27.24	27.22	0.04
Lead	0.92 to 1.00^b	0.96	0.97	0.02
Iron	0.16 to 0.19	0.18	0.17	0.01

[a] Excluding 1 result of 26.96.
[b] Excluding 1 result of 1.05.

TABLE VI.—BRONZE.
First Results Reported by 8 Analysts.

Constituent	Spread of Results, per cent	Mean, per cent	Probable Value, per cent	Average Deviation from Probable Value, per cent
Copper	61.45 to 61.56^a	61.51	61.51	0.02
Manganese	1.49 to 1.56	1.51	1.51	0.02
Aluminum	0.90 to 0.96	0.93	0.93	0.02
Tin	0.82 to 0.85	0.83	0.85	0.01

[a] Excluding one result of 61.38.

TABLE VII.—ALUMINUM ALLOY.
First Results Reported by 8 Analysts.

Constituent	Spread of Results, per cent	Mean, per cent	Probable Value, per cent	Average Deviation from Probable Value, per cent
Manganese	0.55 to 0.59^a	0.57	0.57	0.01
Silicon	0.42 to 0.47	0.45	0.46	0.02
Magnesium	0.39 to 0.42^b	0.41	0.41	0.01
Iron	0.376 to 0.42	0.40	0.40	0.01

[a] Excluding one result of 0.6 3.
[b] Excluding one result of 0.49.

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[8] 1939 Book of A.S.T.M. Standards, Part I, p. 541.

[9] *Ibid.*, p. 545.

in 1000; he is doing very exceptional work if he reaches 1 part in 10,000; and he should be employed in subatomic weight determinations if he can work to 1 part in 10,000,000.

The precision and the accuracy that are obtained in determining components of typical materials are illustrated in Tables II to VII. These are based on results that were reported by analysts who cooperated with the National Bureau of Standards in the standardization of the materials in question. It should be borne in mind that analyses were made on samples which had been prepared in such a way as to insure homogeneity, that the analyst used methods which he considered adequate and no doubt took special care in his tests, and that each reported result probably represents the average of at least two satisfactory determinations. The indicated errors therefore represent what can be expected in special rather than in routine work.

CHANGES IN CHEMICAL ANALYSES AND CHEMICAL REQUIREMENTS

Following the advent of standard methods and standard samples, have come changes in chemical analysis and chemical requirements involving more complex materials, more complete analyses, determinations of constituents occuring in very minute amounts, demands for greater accuracy in many of the determinations, and greater speed in analyzing.

Naturally occuring materials such as silicate rocks are as they always have been, save for the ravages of time. The same cannot be said of man-made materials. Witness the changes that have taken place in common materials such as paints, ferrous and non-ferrous metals, and in ceramic materials.

The pigments in a ready-mixed outdoor house paint may now be a mixture of lead, titanium, zinc, magnesium and calcium compounds instead of the old-time basic lead carbonate. Many irons are no longer plain; alloy steels may now contain selenium, columbium, tantalum, titanium, lead, and boron in addition to the now commonplace alloying elements, nickel, chromium, vanadium, molybdenum, and tungsten. Non-ferrous metals appear in new compositions containing tellurium and beryllium, while some of the porcelain enamels might be called the ceramic sink. In addition to the planned changes in composition there must also be considered the changes that will ensue as more and more scrap finds its way into production. In view of the nonselectivity of the analyst's methods of determination, each new constituent must be closely scrutinized for its probable or possible behavior in the methods that are in use. Not so long ago, the advent of high-sulfur steels played hob with the standard methods for carbon that were in use, and necessitated the introduction of entirely new safeguards.

The trend toward more complete analyses can be illustrated by the increasing number of elements certified in the Bureau's certificates of analyses of its standard analyzed samples. For example, in cast irons the number doubled from 6 in 1906 to 12 in 1928, copper, nickel, chromium, vanadium, molybdenum and arsenic having been added to the elements manganese, phosphorus, sulfur, silicon, titanium, and carbon in its two forms, which were originally certified. In plain carbon steels, the five elements carbon, manganese, phosphorus, sulfur, and silicon which sufficed at the turn of the century were long ago augmented by copper, nickel, chromium, vanadium, molybdenum, and arsenic as a matter of course, and sometimes by others such as tin, nitrogen, aluminum and alumina. There is a similar tendency in the non-ferrous field, as illustrated by the Tentative Specifications for Aluminum-Base Alloy Sand Castings (B 26 – 41 T)[10] which list, in addition to aluminum, copper, iron, silicon, magnesium, manganese, zinc, titanium, nickel, chromium, and "total other impurities."

Not so long ago a reported "trace" was acceptable in chemical analysis, and was commonly interpreted as denoting an amount less than 0.01 per cent of the material. The practical reason for this limit was the general use of 1-g. samples, and the difficulties in accurately determining amounts as small as 0.1 mg. by the methods then employed. Today, large tonnages of materials are sold and accepted on chemical requirements far below the old "trace" limit, as for example the 0.007 per cent maximum for lead and the 0.005 per cent maximum for iron and cadmium in the Standard Specifications for Slab Zinc (Spelter) (B 6 – 37)[11]; the 0.0015 per cent maximum for silver, copper, arsenic, and zinc in Tentative Specifications for Pig Lead (B 29 – 40 T)[12]; and last and least the chemical requirements such as "none" for zinc and aluminum in Standard Specifications for Solder Metal (B 32 – 21).[13] The last is a compliment to the analyst, for it implies supernatural powers of detection on the material, or of clairvoyance as to what was the metallurgist's thought on the mental side. In truth it is usually a confession on the part of the metallurgist that he has no metallurgical or analytical data which will enable him to set a definite limit. That the metallurgist has some misgivings is indicated in the Tentative Specifications for Copper-Base Alloys in Ingot Form for Sand Castings (B 30 – 41 T),[14] in which the term "none" as applied to the aluminum content of some of the alloys is defined as "0.005 per cent when determined on a 10-g. sample." What would happen if the analyst found 0.01 per cent on a 1-g. sample is a problem.

Chemical requirements of "none" are on a par with the maximums for "Other Impurities," or for "Total Other Impurities" which still obtain in some specifications. If the analyst takes these requirements seriously and starts searches there is no limit to what he may uncover. It is the exception to find less than a dozen elements in any metal. Some of these are of common types that might be expected, some are of uncommon types like gallium in aluminum or germanium in zinc, that may be unsuspected, and others, like silver in most metals, are not normally considered in the environment in question. Some materials, as for example clays and phosphate rock, may show as many as one third of the known elements.

It is sometimes asked "What is the analyst's limit in his searches for minor constituents?" The answer depends on the nature of the task and is ultimately governed by the extent to which the analyst can exclude the substance that is sought from his reagents, reaction vessels and extraneous sources. Compositions of the National Bureau of Standards Standard Analyzed Samples of Spelter are certified to percentages as low as one ten thousandth (0.0001) for arsenic, eight one hundred thousandth (0.00008) for silver, and four one hundred thousandth (0.00004) for manganese. The last represents one part in 2.5 millions and is below the limit of detection by direct spectroscopic test in this material. It is safe to say that a "basement" of 1 part in a million (or 0.0001 per cent) was passed long ago.

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[10] 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 314.

[11] 1939 Book of A.S.T.M. Standards, Part I, p. 680.

[12] 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 438.

[13] 1939 Book of A.S.T.M. Standards, Part I, p. 669.

[14] 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 376.

Only last month we read[15] that for the reliable operation of electronic tubes there is required mercury of a purity of 99.99995 per cent (one part of impurity in two million parts of mercury); material conforming to this requirement is being produced at the rate of half a ton per day.

For determinations of these minor constituents the growing use of spectrographic methods has been a welcome development. It must be said here, however, that the analyst has learned that caution must be used in interpreting the term "spectroscopically free." In testing material "as is," as for example the detection of manganese in the spelter just cited, the chemical analyst has outlasted his brother spectroscopist often enough when reactions are favorable, to demonstrate that both have limitations which some are loath to admit. In tests following separations and concentrations, the spectroscopist will, of course, outlast the analyst.

The time alloted for chemical determinations or analyses in industry of today is a far cry from what was considered absolutely necessary even at the beginning of the century.

This has been a natural result of the large-scale manufacture of materials which must meet rigid chemical requirements, and which therefore require constant checking from the raw material to the finished product by methods that will not delay rapid movement during the processing. Oftentimes, as in operations involving the electric furnace, each minute's delay on the part of the laboratory is watched.

The gradual shortening of the time allowed for such analyses has naturally speeded the search for methods that yield results of satisfactory accuracy within the shortest possible time. This calls for the paring of time-consuming operations to a minimum. So in wet methods for manganese we have seen the passing of the gravimetric phosphate method in favor of the more rapid volumetric bismuthate filtration-titration method, the omission of the filtering operation through the substitution of the volumetric persulfate titration method, the by-passing of the titration bottleneck by the adoption of the spectrophotometric methods for manganese, and finally the abandoning of wet methods entirely in favor of direct spectroscopic test on the sample itself.

The illustrations, which have had to do with the steel industry, might be multiplied to cover any industry where chemical requirements are in question or manufacturing techniques require constant control. In the glass industry, for example, the urge for more rapid methods comes more from the need of closer control of the manufacturing operation than on chemical requirements.

Many do not appreciate the advances that such evolution represents, often with an increase of accuracy as well as a saving of time. To those who do not we might call attention to the old wet-combustion method of the nineties for carbon in steel, with its 3 to 4 hours of elapsed time and uncertainty of results, as compared with modern dry combustion methods which require less than 10 minutes and yield results that are much more dependable and accurate. How far we have come may be indicated by observations made 53 years ago by Dr. Langley, who said in an address on International Standards for the Analysis of Iron and Steel: "The analytical chemist is expected to arrive at results in eight or ten hours, which, if they could have been done at all twenty years ago, would have demanded one or two weeks of labor and even then would have been far less accurate." Today, the time required for many of these analyses comes closer to 8 to 10 minutes than to 8 to 10 hours, and the results must be more accurate.

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[15] *Chemical and Engineering News*, Vol. 20, p. 665 (1942).

The analyst can claim with pride that the shortening of the time required for chemical analyses has played no small part in the evolution of our manufacturing processes.

Whenever chemical requirements are inserted in specifications the methods of analysis that are used to check them are sure to receive policing, and faulty or inaccurate methods sooner or later go into limbo. The stiffer the chemical requirements the stiffer the examination must be. In such case, the urgent consideration is that lack of accuracy may mean rejections of material, financial loss, or even the loss of the analyst's job itself. It is quite safe to say that chemical requirements have had more to do with improving the accuracy of chemical analyses than any other single factor since the beginning of the century.

The demands for more accurate analyses can be illustrated by the differences between the chemical requirements for manganese bronze in 1939 and in 1911. The percentages given in 1911 are expressed in 1 or 2 significant figures, as for example copper 55 to 60 per cent, tin not over 2 per cent, and manganese not over 0.5 per cent. In 1939, the percentages are expressed in 2 to 4 significant figures, as for example copper 56.00 to 65.00, tin 1.00 per cent maximum, and lead 0.20 per cent maximum. One difficulty with such changes is that the analyst is often in doubt as to whether the change is one of metallurgical or editorial policy. In other words, whether analyses should be rounded off to the second decimal place and, if so, whether an indicated percentage of 55.99 as against the 56.00 minimum for copper or of 0.21 as against the 0.20 maximum for lead would be cause for rejection. It is not difficult for the analyst to check the lead requirement, which calls for accuracy no better than 1 part in 20. To check the copper requirement, however, calls for accuracy to better than 1 part in 5000, which is severe in ordinary analytical work.

It is comforting to note that a start toward clarification has been made in the Tentative Specifications B 26 – 41 T[10] which carry a clause under the chemical requirements to the effect "For purposes of acceptance and rejection, an observed value or a calculated value obtained from an analysis should be rounded off to the nearest unit in the last right-hand place of figures used in expressing the specified limit."

To meet the increasing demands on the analyst there have, of course, been numerous developments in methods of chemical analysis which can be given but passing mention here. These have included improved methods of separation, more selective reagents, the growth and improvement of volumetric methods, the development of colorimetric, spectrophotometric and nephelometric methods, the rise of microanalytical methods, and the increasing use of physical and chemico-physical methods such as those based on the use of the spectroscope and polarograph in the general field and of special methods such as the electrical resistance method and the magnetic method for carbon in steel in special fields.

Two developments deserve special mention, the rise and importance of spectrographic procedures in qualitative and quantitative analysis in the inorganic field, and of microanalysis in the organic field.

It can be truly said that the growing use of physical and chemico-physical methods has come to the rescue of routine chemical analysis, and has prevented it from becoming completely swamped by the demands of our present-day industrial set-up.

I have outlined the main problems of our chemical analysis of today. As for the future, I picture a chemical analysis in which the analyst has bequeathed the drudgery of the routine determinations of today to the physicist and the physical chemist, and has come back to his own, the establishing of fundamental values in the Sciences and the Arts.

ANNUAL REPORT OF THE EXECUTIVE COMMITTEE

Unquestionably there is one paramount theme which needs to be discussed, even if briefly, in this report to the Society from the Executive Committee, namely, the manner and extent to which the Society is contributing to the war effort.

This is the second time in its history as an incorporated Society that A.S.T.M. has been in a war period, but the contrast between the extent of the Society activities now and in 1917 is so great and indeed the present emergency situation is so much more complicated than it was 25 years ago, that there have been few precedents which could be followed. One basic policy was immediately established and has been and will be adhered to, namely, to make available all of our facilities where they can be used to advantage in assisting the Government and industry in coping with the many intricate problems which have arisen in the field of engineering materials. It had been apparent for many years that the next general war would be basically a war of materials, and that therefore the research and standardization activities in A.S.T.M., dealing as they do with materials, would offer many opportunities for important contributions by the Society to the winning of the war.

More than two years ago it was increasingly apparent that the United States might soon be involved in a global war and some of our activities at that time were intensified—as an example, the activities of Committee B-5 on Copper and Copper Alloys which at that time began to accelerate its very constructive work in numerous problems of particular importance to the Army Ordnance. More than a year ago, as indicated in the 1941 Annual Report of the Executive Committee, A.S.T.M. offered its assistance to various agencies of the Government in the realization that much of our effort as a national technical society should be of help.

There has hardly been any field of materials which has not been markedly affected by the war and consequently many branches of A.S.T.M.'s work have been influenced by the emergency situation. Probably the greatest effect has been in the field of ferrous and nonferrous metals—yet there are many nonmetals fields where A.S.T.M. has rendered notable emergency service. While virtually impossible to delineate, at least in any reasonably condensed report, all significant phases of the Society's activities that have contributed to the war effort, reference should be made to certain general matters and to a few selected cases which are representative of the types of work our committees are doing.

The Executive Committee devoted a considerable portion of its two-day January meeting to a full discussion of problems incident to the war and how the Society through its research and standardization work in materials, and related activities, could best assist in the war effort. Almost every phase of A.S.T.M. work came in for comment and scrutiny.

There was unanimous agreement that research leading to the development of new products and uses and more authoritative information on properties of materials would be one of several important cushions needed to lessen the post-war impact. It was generally agreed that one very important contribution the Society could render would be to intensify its standardization activities, leading to the publication as soon as possible after the annual meeting of a complete combined 1942 Book of A.S.T.M. Standards. The tremendous increase in production has focused attention as no other factor could on sound and authoritative specifications. At no other time in our history as a nation have there been so many technical men working on specifications and so much intensive cooperative effort devoted to agreeing on standards—many of them of an emergency nature.

A procedure was established by the Executive Committee for handling emergency alternate provisions in specifications and consideration was given to the best ways to handle expeditiously the issuance of complete emergency specifications or test methods—these various procedures are outlined in another portion of this report. Reference is made in succeeding paragraphs to a number of committees which have found it desirable to issue emergency alternate provisions which can be invoked by the buyer in the interest of procurement or conserving critical or strategic materials. Unquestionably, the work of the standing committees in developing these emergency items has been one of the outstanding developments during the year and as changes take place in the field of engineering materials, other emergency changes will be forthcoming.

The Report of Committee E-10 on Standards includes a list of all emergency provisions and complete emergency specifications issued up to the present time. All emergency items are issued in the form of pink slips or complete specifications and the provisions are also published in the ASTM BULLETIN. Emergency specifications have been made available to all members on request and a complete set of the emergency provisions was mailed to the members early in June. Several important groups, among them Government and War Production Board officials, received copies of those in which they were specifically interested shortly after the material came off press.

Research and the War Emergency.—There is no question that both fundamental and applied research have contributed immeasurably in helping to make the United States a leader in materials technology, whether this applies to the pursuits of peace, or the conduct of a war. Certainly there are numerous problems which have arisen during the past year which could only have been solved by competent research staffs and the cooperative efforts of organizations which have had research under way. Research to develop a sound basis of specification values or sound testing procedures is fundamental in our standardization work.

During preparations for national defense and since the war began, the Society on numerous occasions has been asked to assist in problems relating to materials entering into ordnance or products essential in the war effort. In a number of instances A.S.T.M. committees have had to initiate promptly and to carry through with the least possible delay research projects to develop authoritative information on which decisions could be based.

An example of this is the work which resulted in the emergency standard ES-2, providing specifications for hot-dip lead coating on hardware. The necessity of conserving other metallic coating materials led Committee A-5 on Corrosion of Iron and Steel to draft promptly proposed requirements for lead-coated hardware. Concentrated research work carried out at the Commonwealth Edison Co., Bell Telephone Laboratories, Inc., and Western Electric Co., resulted in data on which the requirements are based. The prompt promulgation of this urgently needed specification exemplifies the effective way in which our standing committees can work and the importance of close cooperation all along the line.

Much A.S.T.M. work has been effective in promoting a sound use of products through establishment of specifications based on extensive research—for example, die castings. The work of Committee B-6 on Die-Cast Metals and Alloys has been outstanding, and it was but natural that when consideration arose of possible steps to conserve aluminum, this committee should be asked to make recommendations. As a result several emergency grades investigated in cooperative research work have been suggested and have been set up as alternates in the Specifications for Zinc-Base Alloy (B 86) and the Aluminum-Base Alloy (B 85) Die Castings. An important point brought out in this work was that for many uses zinc-base die castings with drastically reduced aluminum content could not be successfully made.

In view of the pressing need for satisfactory information on hardness conversion relations of cartridge brass, the Section on Indentation Hardness of Committee E-1 on Methods of Testing, which is investigating hardness characteristics of various materials, concentrated its first efforts on cartridge brass and there was issued an emergency standard ES-4 which gives relations between diamond pyramid hardness, Rockwell hardness, and Brinell hardness. This work is important not only because of the tremendously increased production of this material for ordnance, but also in lend-lease problems. Round-robin tests were conducted at five cooperating laboratories and when the test data as compared with experience data checked satisfactorily the committee felt its proposals were sound.

The necessity of conserving nickel for the most important military and naval uses was apparent many months before a state of war existed and in June, 1941, Committee B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys was requested to study and make prompt recommendations concerning conservation of nickel in alloys in this field. The committee, having before it the results of earlier studies made at certain laboratories, quickly agreed on a report, which was summarized in the August, 1941, ASTM BULLETIN and forwarded directly to the Office of Production Management. This has been effectively used. This project demonstrates a somewhat different type of research activity where the experience and judgment of the committee members counted very heavily in reaching conclusions and where no actual research had to be undertaken. It is a splendid illustration of how the technical knowledge of materials men serving in A.S.T.M. can be used effectively.

The current report of Committee E-9 on Research contains an excellent discussion of policy that should guide A.S.T.M. research activities under war conditions.

Specifications and the War Emergency.—The production of materials first for defense, and then for war, in such huge quantities has emphasized as no other situation could the importance of satisfactory specifications. At the same time large increases in quotas expected from certain industries have stressed the necessity of some relief during the emergency with respect to certain testing procedures. Scarcity of certain strategic or critical materials, with necessity of conservation, has influenced to a great extent many standardization activities. It is truly inspiring to note with what promptness A.S.T.M. standing committees drafted emergency provisions or complete specifications or considered emergency situations which affected requirements of a standard. While the reports of the standing committees detail these activities, it seems in order to indicate certain fields where emergency activities have been particularly outstanding.

Of the some 50 specifications affected by emergency alternate provisions, 19 are in the jurisdiction of Committees A-1 on Steel and A-10 on Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys. Our conduct of the war, based as it is on ships, guns, shells, and similar products where steel is essential, is related directly to the production of this basic commodity and the remarkably successful efforts of various units in the steel industry in stepping up production are a matter of record. In no other industrial fields are A.S.T.M. specifications more widely used or more influential and our Steel Committee has labored intensively to keep its specifications up to date.

The scarcity of certain elements in relation to inordinate war production demands, has made all possible conservation essential. Many of the emergency provisions developed in Committee A-1 have been in this interest such as certain changes in the chemical composition of steel castings, and spring wire, and in the field of rails and track fastenings. The committee has also set up emergency provisions so that, for example, a heat of casting steel made to a certain A.S.T.M. specification would at the same time meet Federal or Navy requirements.

There are many outstanding illustrations in the work of our non-ferrous metals committees involving emergency standards activities, such as the prompt development by Committee B-1 on Copper and Copper-Alloy Wires for Electrical Conductors of a complete emergency specification ES – 1 covering lead-coated wire for electrical purposes which was developed to conserve other protective coating elements of a more critical nature.

The crucial tin situation resulted in orders from the War Production Board forbidding the use of bearing alloys with more than 10 per cent tin, or solder metal which had more than 30 per cent tin. To help in this critical situation, A.S.T.M. Committee B-2 on Non-Ferrous Metals and Alloys, with the cooperation of the Society of Automotive Engineers and War Production Board personnel, agreed on a number of emergency alloys which were set up as alternates in two A.S.T.M. specifications covering White Metal Bearing Alloys (B 23) and Soft Solder Metal (B 32).

The emergency work of Committee B-5 on Copper and Copper Alloys, Cast and Wrought which, as indicated above, began its series of constructive studies and actions more than two years before war began, has been extremely important—the latest developments relating directly to the war effort involve the copper-base alloy castings field where several emergency alloys have been agreed on to conserve tin or copper or both. The May ASTM BULLETIN contained a rather extended discussion of this work. Many other phases of the work of Committee B-5 have been very helpful in the war effort.

A very recent development in the non-metals field is the promulgation by Committee C-1 on Cement of Emergency Alternate Specifications for Portland Cement EA – C 150 which have just been approved (June 6). The War

Production Board requested the Society to submit recommendations for limitation of types of cement to be produced and any other modifications in specifications that would contribute to increased production and conservation of cement for war work and as a result these specifications have been prepared.

Because of the extremely critical rubber situation Committee D-11 on Rubber Products has had serious problems confronting it and the several emergency alternate provisions listed in its 1942 report reflect this situation. The A.S.T.M. standards in this field have been widely used and the actions taken by the committee exemplify the desires of the Society's technical groups to render all possible assistance. This committee was responsible for the first emergency alternate provision issued by the Society on August 26, 1941, modifying requirements on sheeting in the Standard Specifications for Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound (D 27 – 41) so that the cable manufacturers could have some relief in obtaining one of the most widely used types of sheeting.

Emergency work in Committee D-12 on Soaps and Other Detergents is another case where a standing committee felt it most desirable to assist in effecting savings in critical materials. Emergency alternate provisions were developed in a number of specifications.

Mention was made in the 1941 Executive Committee Report of the important work under way in Committee D-20 on Plastics. Increasing committee activities have resulted in bringing forth a number of new tests as detailed in its latest report.

Miscellaneous War-Effort Activities.—In line with the objective of making the ASTM BULLETIN of maximum benefit to members during the war emergency, special articles were included in the three issues published this year—January, March, and May—with condensed information giving the set-up of the OPM and WPB (a special four-page article with charts outlined the various divisions of the War Production Board, directors, branch chiefs and related information). Efforts are made to give promptly news of committee activities which relate to the war effort. Lists of the Federal Emergency Specifications have been published in the BULLETIN and the Society has offered to send complete copies of these specifications to members. A large number of requests have been received. The development of these emergency specifications and their promulgation, in which efforts the WPB Bureau of Industrial Conservation is most active, are of vital importance in conservation of critical and strategic materials.

Many members of the Society are spending all their time in the war effort, some in the armed services and many more in connection with production of war materiel; there are also many Society members who are serving in the War Production Board and other Governmental agencies, and in various advisory capacities. Many members of the Society's Committee A-1 on Steel are serving on the technical advisory committees that are developing the lists of National Emergency Steel Specifications for the War Production Board.

As the Executive Committee advised a year ago, the services of the Secretary-Treasurer were loaned to the War Production Board (then Office of Production Management) on a part-time basis beginning April, 1941, with the understanding that his responsibilities to the Society would need to be maintained and that the Executive Committee reserved the privilege of reviewing the situation periodically. Such review during the past year has led the Executive Committee to assent to the desire of WPB that the arrangements be continued, so that the Secretary-Treasurer has spent over a year in the War Production Board, devoting about three-fourths of his time to the work. He is now Chief of the Specifications Branch, Bureau of Industrial Conservation; also administrator of the National Emergency Steel Specifications project of the WPB, sponsored by the American Iron and Steel Institute, Society of Automotive Engineers, and A.S.T.M. with the collaboration of the War and Navy Departments.

Committee Activities:

As would be expected, all the standing committees have re-orientated their activities having the war effort particularly in mind. Since materials are playing such an important role, it is especially important that specifications covering the critical materials be reviewed and kept up to date; also that attention be given to development of information with respect to these materials. This is reflected in the reports of many of our standing committees being presented at the annual meeting this year, particularly those in the metals field.

This general situation has also received careful consideration on the part of the Executive Committee, namely, as to how the standing committees can best function in the war effort and how their activities should be carried on under war-time conditions. As a result of discussions in the Executive Committee at its January meeting a letter was addressed to the officers of the standing committees reviewing the situation and emphasizing the possibilities of a procedure that had been established for the development of emergency alternate revisions; also the handling of requests for information and advice from either the Government or industry. The Executive Committee also inquired concerning the plans of the individual standing committees covering their future activities in the light of present conditions and asked the committees to suggest aspects of their work that should be taken into account in fixing Society policies. The responses indicated a universal desire on the part of the standing committees to be of every assistance possible to the Government and industry at large in the war effort. A number of the committees felt that the best contribution would be a concentration on emergency modifications of existing standards so as to implement War Production Board orders and in general to have specifications in line with limitations on strategic materials. Other committees discussed principally the research activities before them, while some indicated the part that could be played by committees in an advisory capacity.

As a result of these discussions the following suggestions have been made to the standing committees as a guide in their future work:

1. In view of the all-importance of time, all of our committee standardization activities that would be of definite assistance to the war effort should be expedited.
2. There is need for faster test procedures.
3. Whether or not research work should be carried on depends in large part upon its relation to the war effort. Work that is already in progress should be pushed to the nearest suitable end-point where its stoppage will not mean the loss of valuable data and the waste of time and materials. Work which has just been inaugurated should be scrutinized carefully to determine to what extent it will be of assistance to the war program and whether the expenditure of the necessary time, energy, materials, and money is warranted under present conditions. Research work which has not been started should also be reviewed on the latter basis.
4. Results of research work should be

carefully scrutinized prior to publication, to determine whether such publication would be of aid and comfort to the enemy.

The current reports of Committees E-9 on Research and E-10 on Standards deal with certain phases of the committee work and, supplemented by reviews that will appear in the ASTM BULLETIN, give a general summary of the committees' activities. This report, therefore, makes brief reference only to several administrative actions during the year.

In response to several suggestions that the Society undertake the development of methods of determining hardenability of steel, Committee A-1 on Steel was asked to undertake this project. A new subcommittee organized for this purpose began its work promptly and a proposed Tentative Method of End Quench Test for Hardenability of Steel (A 255 – 42 T) has been approved by Committee E-10 on Standards.

As an outcome of specification work on welding rods in the American Welding Society and Subcommittee XXI, of Committee A-1, on Steel Welding Electrodes and Welding Rods, the Executive Committee, in collaboration with the officers of the American Welding Society, has decided to organize a Joint Committee on Filler Metal, to be responsible for specification work in this field and to take over the activities of Subcommittee XXI and the A.W.S. Filler Metal Specifications Committee. It is believed that this will provide a better set-up for consideration of the problems involved, particularly since standards for higher alloy steels and nonferrous metals are entering into the picture which will also be of interest to several other A.S.T.M. committees, including some in the non-ferrous field. An organization meeting of the joint committee is planned during this annual meeting.

Based upon discussions that have been taking place during the past few years, both in the individual standing committees dealing with corrosion of metals and subsequently with Committee E-9 on Research, the Executive Committee has agreed to the formation of a new administrative committee, to be designated as an Advisory Committee on Corrosion, which will provide a more effective means of administering the Society's work in the field of corrosion of metals. This Advisory Committee will consist of the chairmen of the six standing committees concerned with the corrosion of metals, namely, A-5 on Corrosion of Iron and Steel, A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys, B-3 on Corrosion of Non-Ferrous Metals and Alloys, B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys, B-6 on Die-Cast Metals and Alloys, and B-8 on Electrodeposited Metallic Coatings; and three members-at-large serving for a term of three years, namely, W. H. Finkeldey, L. W. Hopkins and F. L. LaQue. The committee will concern itself with corrosion investigations, researches, test procedures, etc., in so far as these apply to metals and alloys. An Outline of Scope of Activities and Functions of the committee has been prepared which defines the activities of the committee in such matters as reviewing programs of work on corrosion problems, reviewing reports and investigations, standardizing definitions and test procedures, assuming jurisdiction over corrosion test sites, and acting in an advisory capacity to the Executive Committee and Committee E-9 on Research in various matters. It is expected to hold the organization meeting of the Advisory Committee during the present annual meeting.

Several questions of jurisdiction in the matter of standards have received the consideration of the Executive Committee during the year, one of which was a re-opening by Committee A-5 on Corrosion of Iron and Steel of the decision reported last year that jurisdiction over Tentative Methods of Test for Local Thickness of Electrodeposited Coatings on Steel (A 219 – 40 T) be transferred from Committee A-5 to the newly organized Committee B-8 on Electrodeposited Metallic Coatings. While this transfer was acceptable to Committee A-5 at that time it has since developed that because specifications for this type of coating are under the jurisdiction of Committee A-5, that committee desired joint jurisdiction with Committee B-8 over the methods. Having ascertained that this would be agreeable to Committee B-8, the methods in question have now been placed under the joint jurisdiction of Committees A-5 and B-8.

The question of jurisdiction in the matter of specifications for various nickel-chromium-iron alloy products, in view of certain specifications in this field having been submitted to Committee E-10 on Standards by Committee B-2 on Non-Ferrous Metals and Alloys, was raised by Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys since the material covered would seem to be closely allied to materials that normally fall under the jurisdiction of Committee A-10. This matter was referred to a coordinating committee consisting of representatives of Committees A-1 on Steel, A-10, B-2, and B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys. In view of the relatively low iron content of the products covered, the particular specifications in question were definitely considered to be under the jurisdiction of Committee B-2 and were so assigned. It was agreed that in the future any specifications in this general field developed by any of the committees should immediately be brought to the attention of the other committees represented on the coordinating committee.

Committee E-10 on Standards has recommended that jurisdiction over Tentative Method of Salt Spray Testing of Non-Ferrous Metals (B 117 – 41 T) be transferred from Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys to Committee E-1 on Methods of Testing inasmuch as the scope of the tentative method is being expanded to apply to the testing of organic preservative coatings and the method is also of interest to several other committees. This matter is now being discussed with the standing committees concerned looking toward the proposed transfer of jurisdiction.

Changes in scopes of Committees B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys, D-20 on Plastics and E-4 on Metallography have been approved by the Executive Committee at the request of these committees. The revised statements of scope, with the modifications shown in italics, are, respectively, as follows:

Committee B-4:

"Committee B-4 has jurisdiction over the classification, specification requirements, methods of test, questions of utility and related subjects pertaining to wrought and cast metallic materials and parts for electrical heating and electrical resistance, for the structure and containers of electric furnaces, thermostatic metals and alloys, metallic materials for vacuum tubes and incandescent lamps, *and materials for electrical contacts.*"

Committee D-20:

"The development of test methods *and specifications* applicable to the finished products (molding materials, sheets, tubes and rods, and molded or fabricated articles) in the field of plastics. The scope does not include electrical tests, and materials used in the paint industry and rubber industry, which are covered by other committees."

Committee E-4:

"Methods and technique of examination of all metallic substances by microscope and X-ray diffraction; methods and standards of measurement of grain sizes of *and inclusions in* metals and alloys; methods and recommendations for photography as applied to metallography; methods of thermal analysis of steels; definitions of terms relating to metallography."

Emergency Standardization Procedures:

Since the last annual meeting considerable thought has been given in the Executive Committee and Committee E-10 on Standards to the question of temporary modification of A.S.T.M. standards and the establishment of new standards to meet the war emergency, and while the procedure adopted has been published in the ASTM BULLETIN and the Year Book it is included here in its present form as a matter of official record, particularly since the procedure has been somewhat extended so as to apply to completely new emergency standards.

PROCEDURE FOR EMERGENCY PROVISIONS AND STANDARDS

Emergency Alternate Provisions are issued by the Society in the interest of expediting procurement or conservation of materials during the period of National Emergency. They are intended for use where they may be considered by the purchaser of the material as a permissible alternate for the specific application or use desired.

Such emergency alternate provisions shall first have the approval of the appropriate subcommittee of the sponsoring committee or duly appointed subgroup of that subcommittee and shall have the endorsement of the chairman of the main committee. The emergency provisions shall then be submitted to Committee E-10 for approval for publication. If approved they shall be published with the specification either in the form of a sticker or as an accompanying sheet and shall also be published in the next succeeding issue of the ASTM BULLETIN. Any emergency provisions approved during the year shall be recorded in the next annual report of the standing committee. All such provisions are subject to annual review and the standing committee shall annually report its recommendations with respect to them.

A.S.T.M. standing committees may also find it appropriate to issue complete A.S.T.M. emergency standards which in many instances may be alternates to existing specifications, which latter specifications will be suitably marked. The procedure to be followed by the committee in the development of such emergency specifications shall be the same as that indicated above for the approval of emergency alternate provisions.

The annual report of Committee E-10 on Standards contains a detailed statement of the emergency alternates and emergency specifications that have been issued to date.

In addition to the matter of emergency standards, consideration has been given to several procedural questions in connection with the conduct of committee activities under war conditions. One important deviation from regular practice that has resulted is the authority that has been given to standing committees, who have found it impossible to hold meetings for the purpose, to act by letter ballot on the adoption as standard of existing tentative standards and tentative revisions of standards, subject to confirmation at a meeting of the committee to be held during the annual meeting.

Another important change in procedure is the modification that has been made in the Regulations Governing Standing Committees covered in the following section.

Modification of Regulations Governing Standing Committees:

The Executive Committee and Committee E-5 on Standing Committees have agreed on the desirability of modifying the procedure under which standing committees may act upon recommendations dealing with standards, so that instead of requiring all actions to be taken only at meetings of the main committee, certain types of recommendations may be referred immediately to letter ballot on the recommendation of a subcommittee. At present some of our committees have the major discussions on standards at meetings of the main committee, but others are so organized that recommendations dealing with standards—either the formulation of a new standard or a revision of an existing standard—are given full consideration in a duly constituted subcommittee so that action on the part of the standing committee at a meeting may become something of a formality. It is expected that the modified procedure will expedite the work of a number of committees, particularly in the case of subcommittee work completed in the interim between meetings of the main committee and, as will be seen from the wording suggested, suitable safeguards have been provided to insure consideration being given to negative viewpoints by requiring that the entire committee be advised of those voting negatively in the subcommittee with the reasons therefor and by permitting committee chairmen to hold recommendations for review at a meeting of the standing committee if any negative viewpoint is expressed in the letter ballot of the standing committee.

The following change in Section 14 of the Regulations Governing Standing Committees is therefore announced, with the intention that it shall become effective at the close of the present annual meeting:

Change the first sentence of Section 14 by the insertion of the italicized words and the omission of the words in brackets:

> 14. Procedure Governing Recommendations on Standards.—*Committee* recommendations [of standing committees] affecting standards shall *normally* be agreed upon [only] at *regularly called* meetings [called for that purpose].

Add the following as a new second paragraph of Section 14 after the present second sentence:

> When recommendations affecting standards have been approved by letter ballot vote in a subcommittee, they may, at the discretion of the chairman of the standing committee, be referred directly to letter ballot vote of the standing committee without authorization at a meeting of the standing committee, provided such recommendations are accompanied by a supporting statement together with the results of the letter ballot vote in the subcommittee, including a list of those voting negatively with the reasons therefor. If any negative viewpoint is recorded in the letter ballot in the standing committee, the chairman, at his discretion, may hold the recommendation for review at a meeting of the standing committee and the recommendation shall be held for such review if a negative vote of 10 per cent or more of those voting is cast.

District Committees:

Even though several of the district committees have not found it possible to sponsor local meetings during the year because of inability to obtain certain speakers whose speaking activities have been much curtailed by pressure of emergency matters, each of the committees has rendered service to the Society through membership promotion work and in other ways. All of these activities are appreciated by the Executive Committee. The district committees occupy an important place in the Society's organization, providing a direct contact through local meetings and other ways with many members who do not find it possible to attend the annual or spring meetings, and generally assisting in the promotion of the Society's interests in the areas where the district committees function.

The Headquarters' staff has cooperated with committees that sponsored local meetings during the year and is prepared to continue this cooperation by assisting in obtaining speakers and handling various details.

The Chicago District Committee is cooperating closely with the local Office of Civilian Defense in connection with the study of specifications for blackout materials.

As announced in the March ASTM BULLETIN, the Detroit District Committee has become an affiliate member of the Detroit Engineering Society.

As noted later in this report, the Executive Committee has accepted an invitation from a group of members in the Buffalo and Western New York area to hold the 1943 Spring Meeting in Buffalo, and a Local Committee on Arrangements is being organized with B. L. McCarthy, Wickwire Spencer Steel Co., W. H. Lutz, Pratt & Lambert, Inc., and T. L. Mayer, Department of Technology, Buffalo Public Library serving as temporary chairman, vice-chairman, and secretary, respectively. Recommendations by this group for the formation of a new district committee are before the Executive Committee for consideration.

It is planned to include in the August, 1942, ASTM BULLETIN announcements of new officers and members of the various district committees.

Meetings:

Since the meetings held during the past year have been described fully in the ASTM BULLETIN, it is sufficient here to cite them as a matter of record with a note of appreciation from the Executive Committee to the numerous members who helped to make the meetings successful.

Because the 1941 Annual Meeting in Chicago, June 23 to 27, was so outstanding, it seems well to include a short note concerning this event. With one of the strongest technical programs yet developed, it also marked a new high in attendance—1553 registrations, with more members, committee members and visitors attending than ever before. The Local Committee on Arrangements, which consisted of the Chicago District Committee together with a number of other active Society members, headed by E. R. Young, functioned splendidly in handling its various responsibilities including the planning of a technical session of local interest (sponsored jointly with the Western Society of Engineers), golf tournament, extensive ladies entertainment program, and particularly the very successful cocktail reception and officers' night, a new feature for the A.S.T.M.

The 1942 Committee Week was held in Cleveland, March 2 to 6, 1942, with numerous Society committees participating. Because of the war emergency no spring meeting with its customary technical symposiums or sessions was held. The total registration for the week was 520, which while smaller than in recent years was considered excellent under the circumstances. The Cleveland District Committee assisted in developing plans for the committee meetings.

The following local meetings were held under the auspices of the respective district committees: in Los Angeles, October 28, 1941, an open forum on the subject "Specifications, Their Importance in the Present Emergency"; in Philadelphia, December 5, 1941, on the subjects "Aircraft Engines," "Aircraft Structures," "Aviation Fuels," and "Emergency Specifications"; in Philadelphia, January 12, 1942, on the subject "Fatigue Testing Applied as a Materials Acceptance Test" with a colored movie "America the Beautiful"; in Pittsburgh, March 30, 1942, on the subjects "Rubber and the War," "Gasoline and the War," and "Materials Standards and the War"; in Philadelphia, April 30, 1942, on the subjects "Textiles," "Rubber," and a colored sound movie entitled "Unfinished Rainbows"; in Philadelphia, May 15, 1942, a joint meeting sponsored by the Philadelphia District Committee and the Department of Physics of the University of Pennsylvania on the subject "Physics of Pigments and Glasses," with three technical sessions. Accounts of each of these meetings have appeared or will appear in the ASTM BULLETIN.

It will be noted that the Philadelphia District Committee under the chairmanship of F. G. Tatnall has been particularly active. Each of the four meetings it has sponsored has been very successful, and the Executive Committee is appreciative of the intense interest of this group.

In connection with these local meetings, it is pertinent to note that each committee sponsors a meeting with a definite objective in mind. While there are occasional critical comments on the large number of meetings sponsored by local sections and chapters of various societies, it seems obvious that the occasional meetings sponsored by the A.S.T.M. district committees in their localities should have a very desirable effect in knitting together the members in the particular area, especially when there is a program of an educational and instructive nature, as is usually the case.

The Executive Committee announces that the 1943 Spring Meeting and A.S.T.M. Committee Week will be held next March in Buffalo, N. Y. Because of uncertainties incident to the war emergency no decision has as yet been reached concerning the location of the 1943 annual meeting or whether an Exhibit of Testing Apparatus and Related Equipment, which has been held since 1931 at two-year intervals, will be held next year.

Publications:

The magnitude of the publication activities during the past year has increased somewhat over the previous year due primarily to the Book of Standards Supplements being considerably larger than in the previous years, and also due to the fact that three additional compilations of standards have been established, sponsored respectively by Committees B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys, B-5 on Copper and Copper Alloys, Cast and Wrought, and (jointly) C-9 on Concrete and Concrete Aggregates and D-4 on Road and Paving Materials. Added to this has been the necessity of handling as a special publication matter the printing as stickers of some 50 Emergency Alternate Provisions in existing standards and 6 complete new Emergency Standards.

A publication worthy of special comment is the X-ray Diffraction Data Cards. This comprises the printing of some 4000 cards, containing data on the X-ray diffraction constants of some 1300 compounds; 250 sets of the cards have been printed. This index is of considerable utility in identifying these compounds after the X-ray diffraction constants have been determined.

Another publication of interest is the Cotton Yarn Appearance Standards, comprising twenty $5\frac{1}{2}$ by 10-in. photographs representing four grades each of five groups of yarn numbers. They were formerly published by the Department of Agriculture, but the Society was asked to reproduce the charts and take over their distribution.

The demand for the special compilations of standards continues to grow, as evidenced by the following list:

A.S.T.M. Standards on Electrical-Heating and Resistance Alloys (Committee B-4), 111 pages (plus 39-page paper laid in the compilation), 750 copies, 18 standards, first issue of compilation, will probably be published annually.

A.S.T.M. Standards on Copper and Copper Alloys (Committee B-5), 360 pages plus 1 EA sticker and 1 insert plate, 1200 copies, 82 standards, first issue of compilation, will probably be published annually.

A.S.T.M. Standards on Cement (Committee C-1), 103 pages, 2000 copies, 10 standards, published annually.

A.S.T.M. Standards on Mineral Aggregates (Committees C-9 and D-4), 140 pages, plus 1 insert color plate, 1000 copies, 50 standards, first issue, will probably be published annually.

A.S.T.M. Standards on Petroleum Products and Lubricants (Committee D-2), 406 pages, 5000 copies, 83 standards, published annually.

A.S.T.M. Standards on Electrical Insulating Materials (Committee D-9), 460 pages, plus 1 insert plate, 750 copies, 59 standards, published annually.

A.S.T.M. Standards on Rubber Products (Committee D-11), 288 pages, plus 1 EA sticker, 1200 copies, 39 standards, published biennially

A.S.T.M. Standards on Textile Materials (Committee D-13), 399 pages, 1600 copies, 69 standards, published annually.

Selected A.S.T.M. Standards for Students in Engineering, two printings: first printing, 240 pages, 3000 copies, second printing, 248 pages, 6000 copies—37 standards.

The following regular publications were issued and distributed to the members:

1941 *Proceedings*, 1388 pages, plus 1 insert plate, 5000 copies. In addition preprints of reports and papers totaled 1309 pages, 1600 copies.

1941 Supplements to Book of Standards:
- Part I, 619 pages, plus 1 EA sticker and 2 insert plates, 6850 copies.
- Part II, 449 pages, 5450 copies.
- Part III, 663 pages, plus 1 EA sticker, 5100 copies.

Index to Standards Including Tentative Standards (1941–1942), 196 pages, 17,000 copies.

Year Book, 336 pages, 3500 copies.

ASTM BULLETIN, six issues, August, 1941, to May, 1942, 408 pages, average edition, 6700 copies.

The following symposiums, held at the Washington Spring Meeting in 1941, were published and furnished to members on order:

Symposium on Color, 85 pages, 2500 copies.

Symposium on New Methods for Particle Size Determinations in the Subsieve Range, 117 pages, 2000 copies.

A statement of the volume of the principal publications for the past four years follows. This statement is not truly comparative for 1938 because most of the standards published that year were in single-column format which has now been replaced by double-column style:

Publications	1938, pages	1939, pages	1940, page	1941, pages
Proceedings	2084[a]	1352	1403	1388
Book of A.S.T.M. Standards	....	3794	....	....
Supplements to Book of A.S.T.M. Standards	251	....	1445	1731
A.S.T.M. Methods of Chemical Analysis of Metals	35	256	....	....
Index to A.S.T.M. Standards and Tentative Standards	128	160	172	196
Year Book	286	304	320	336
Book of A.S.T.M. Tentative Standards	1796	(Discontinued as separate publication)		
Spring Meeting Symposiums	57	251	100	202
	4637	6117	3440	3853

[a] Includes new tentative standards and tentative revisions of existing standards, under present plan published in Book of Standards and Supplements thereto.

The following publications were also issued, the first two being reprints from the *Proceedings:*

Report of Committee A-5 on Corrosion of Iron and Steel, 29 pages plus 1 insert table, 500 copies.

Symposium on Problems and Practice in Determining Steam Purity by Conductivity Methods, 84 pages, 3500 copies.

Index to Technical Papers and Reports (1936–1940), 191 pages, 1500 copies.

Impact Resistance and Tensile Properties of Metals at Subatmospheric Temperatures, 112 pages, 1500 copies.

Tentative Specifications for Electrodeposited Coatings (two printings), 23 pages, 1000 copies each printing.

X-ray Diffraction Data File for Chemical Analysis, 3936 cards, 250 sets.

Tentative Method for Evaluating Degree of Resistance to Rusting Obtained with Paint on Iron or Steel Surfaces (D 610 - 41 T), a 3 by 5-in. folder of 16 sections, 500 copies.

Standards for Cotton Yarn Appearance, 100 sets of 20 photographs.

Viscosity Temperature Charts: Chart A, 20,000 copies; Chart C, 5000 copies; Chart D, 5000 copies.

The special Report on Significance of Tests of Concrete and Concrete Aggregates, a revision of the 1935 publication, mentioned in the 1941 report of the Executive Committee, is well under way, and it is expected that the publication will appear sometime in the summer.

The Symposium on Mineral Aggregates, also mentioned in last year's report, has progressed less rapidly, due to the war effort, but work will be carried on as time permits.

Rules Governing the Award of the Charles B. Dudley Medal:

As originally worded the Rules Governing the Award of the Charles B. Dudley Medal limited consideration for the award to papers presented at annual meetings of the Society. Committee E-6 on Papers and Publications has recommended to the Executive Committee the desirability of including also papers presented at the spring meetings and possibly all papers published by the Society. As a result of its consideration the Executive Committee has modified the Rules Governing the Award to provide that all papers reviewed by Committee E 6 on Papers and Publications and published in their entirety by the Society shall be eligible for award of the Dudley Medal.

TABLE I.

Class of Membership	Membership		Losses				Additions		Total		
	June 1, 1941	June 1, 1942	Resignations	Dropped	Deaths	Transfer	Transfer	Election	Loss	Gain	Increase
Honorary	9	7	...	...	2	...	...	...	2	...	2[a]
Perpetuity	9	9	...	...	...	...	...	...	...	...	...
Sustaining	136	148	...	...	...	...	11	1	...	12	12
Company, Firm, etc.	1013	1070	17	9	...	12	3	92	38	95	57
Individual, etc.	3225	3306	87	64	19	3	13	241	173	254	81
Junior	78	100	3	3	...	12	...	40	18	40	22
Total	4470	4640	107	76	21	27	27	374	231	401	170
Student	437	419	20	273	1	16	...	292	310	292	18[a]

[a] Loss.

Membership:

The membership of the Society on June 1, 1942, was 4640, which is the highest in its history. Statistics showing gains and losses in various classes of membership are given in Table I.

The net gain for the year of 170 compares with 156 for the preceding year. Election to membership were 374 exceeding those for the previous year by 4; losses from resignations and delinquencies were 183 compared to 191 for the preceding year. There has been a significant gain in Junior Members from 78 to 100. The number of student members was 419.

The number of sustaining members has been increased by 12 as of June 1, making the total number of sustaining members 148. The names of the new sustaining members are as follows:

Autocar Co., The
Bonney-Floyd Co., The
Chase Brass and Copper Co., The
Crucible Steel Co. of America
General Dyestuff Corp.
Koppers Co., Tar and Chemical Division
Lukens Steel Co.
Medusa Portland Cement Co.
Newport Industries, Inc.
Riverton Lime and Stone Co.
Walworth Co., Inc.
Worth Steel Co.

The United Aircraft Corp. will become a sustaining member shortly replacing the present company membership of the Pratt & Whitney Aircraft Division.

The Executive Committee expresses on behalf of the Society its thanks to these companies for their willingness to lend increased financial support to the Society's activities through this form of membership.

The Executive Committee also expresses its thanks to the District Committees and the many members of the Society who throughout the year have aided so effectively in the acquiring of new members.

As announced a year ago, the Executive Committee has authorized the Secretary-Treasurer to waive upon their request the dues of members of the Society who have entered the armed forces of their country. The names of such members will be retained on the membership rolls during the period of their service and they will retain all privileges of membership, save that the Book of Standards and the *Proceedings* will not be sent them. They will continue to receive the ASTM BULLETIN. Only 15 members of those now in the armed services have requested such waiver of dues. Regarding membership on standing committees of members in service, the standing committees are privileged to take cognizance of any unusual conditions of this kind affecting the activity of their committee members and, if they desire, to place such members in an inactive status for the duration of the emergency.

The Society has lost the following members through death:

	Date of Membership
Bent, H. B.	1907
Calder, Robert James	1914
Cannan, George A.	1928
Coleman, John W.	1923
Davis, John F.	1927
Dunagan, Walter M.	1929
Flannery, William	1930
Fraser, Kenneth	1923
Harrington, John Lyle	1916
Harris, Frank W.	1935
Hayden, H. P.	1923
Jarecki, Alexander	1896
Keegan, Andrew W., Sr.	1940
Lindenberger, Henry	1935
Millsom, J. A.	1935
Norris, George L.	1902
Saeger, Geoff. A.	1922
Sorensen, Louis	1928
Staples, Horace A.	1921
Talbot, Arthur N.	1898
Thompson, Gustave W.	1903

The list of deceased members includes two Past-Presidents and Honorary Members. Arthur N. Talbot, President 1913–1914, and Gustave W. Thompson, President 1928–1929, both served the Society effectively from its early days and contributed largely to its progress. Many of the other deceased members were engaged in the technical work of the Society, George L. Norris having been particularly active in the work of the steel and other committees.

Since instituted by the Executive

Committee in 1938, the Society has awarded certificates commemorating forty years of continuous membership to twenty-eight members. This year fifteen such awards will be made to the following members:

- Charles Derleth, Jr.
- W. C. DuComb
- W. R. Edwards
- J. E. Greiner
- N. F. Harriman
- W. M. Saunders
- H. E. Smith
- Bradley Stoughton
- J. L. Van Ornum
- Ajax Metal Co.
- International Harvester Co.
- Jones & Laughlin Steel Co.
- Lukens Steel Co.
- Sherwin-Williams Co.
- Standard Steel Works Co.

Finances:

Report for the Fiscal Year 1941.—The annual statement of the finances of the Society follows in the report of the auditors for the fiscal year, January 1 to December 31, 1941. The report gives the balance sheet of assets and liabilities, including special and designated funds (that is, the Research Fund, Medal and Lecture Fund, and Committee Funds); statement of receipts and disbursements, in which items are classified into "operating" (budgeted) and "nonoperating"; details of special funds; and investment of Society funds, both book and market values being given.

Total receipts from dues and entrance fees in 1941 were $96,847.07, an increase of nearly $5000 over the preceding year—accounted for by the growth of membership and the continued increase in the number of sustaining members. Receipts from sales of publications totaled $79,434.07 and reflect the continued and increasing demand for the Society's publications, especially the Book of Standards and its Supplements. The receipts from the sale of Selected Standards for Students in Engineering ($3158.27) is the largest for any one year and is due to its wide use in the National Defense Training Courses established throughout the country. Miscellaneous receipts amounted to $23,219.46, consisting principally of receipts from advertising in the ASTM BULLETIN and Index to Standards, receipts from the Exhibit of Testing Apparatus and Equipment, interest and dividends, and registration fees. Receipts from advertising in the BULLETIN, $8910.89, were the highest for any year.

Total operating receipts were $199,500.60. Total operating disbursements with all current bills paid (as detailed in the auditors' report) were $181,497.91, which includes for budget comparison purposes the sum of $6000 applied from current income towards the reserve for publication of the 1942 Book of Standards; also $1600 (one-half of the entrance fees) paid into the principal of the Research Fund and $1471.54 paid into the Employees' Retirement Plan, as well as $733.13, expenses incurred in relation to the Emergency program on national defense. Thus a favorable budget balance between operating receipts and disbursements of $18,002.69 has resulted.

The auditors' report includes a statement of the financial condition of the Society for the past five years. The surplus in the general funds account, exclusive of special and designated funds, is $96,034.51, which compares with $81,144.08 a year ago. In addition, the Reserve for 1942 Book of Standards now totals $13,890.25. The principal of the Research Fund as of December 31, 1941, was $30,391.15.

Investment of Society funds as of December 31, 1941, are given in the auditors' report, both book and market values being shown. The following changes in the investment portfolio are noted:

GENERAL FUNDS

Sold		*Received*
$3000	Wheeling Steel Corp. 1st Mtg. S.F.A. 1966 (called)	$3 150.00
Bought		*Cost*
$4000	General Steel Castings Corp. 1st Mtg. A 5½s, 1949	$3 610.00
3000	Pressed Steel Car Co., Inc. 15 yr. deb. 5s, 1951	2 868.75
3800	U. S. Treasury Bonds 2½ per cent, 1967–72	3 800.00
2000	U. S. Savings Bonds Series G, 1953	2 000.00
25	shares American Can Co., common	2 122.30
6	" American Cyanamid Co. 5 per cent Cumulative Preference (dividend)	......
40	" Westinghouse Electric and Mfg. Co. common	3 456.45
8	" Westinghouse Electric and Mfg. Co. common	560.00
		$18 417.50

RESEARCH FUND

Bought		*Cost*
25	shares Pittsburgh Plate Glass Co. capital stock	$1 955.00

No account has been taken in the accompanying financial statement of the assets of the Society in the form of publications in stock. An inventory of technical publications as of June 1, 1942, may be summarized as follows:

	Number of Copies
Year Book	480
Proceedings	4 731
Index to Proceedings, 6 parts, total	2 408
1939 Book of A.S.T.M. Standards:	
Part I	299
Part II	117
Part III	98
1940 Supplements to 1939 Book of A.S.T.M. Standards:	
Part I	1 300
Part II	834
Part III	879
1941 Supplements to 1939 Book of A.S.T.M. Standards:	
Part I	655
Part II	449
Part III	307
Index to Standards and Tentative Standards	772
Selected Standards for Students in Engineering	7 875
Reprints of Standards and Tentative Standards (approximate)	75 000
Special Reprints (approximate)	31 000

1942 Finances.—The Society's operations for 1942 are based upon budgeted operating receipts of $199,000, of which $185,000 is estimated current receipts and $14,000 is applied to 1942 operations from surplus. Receipts from dues and entrance fees are estimated at $97,500; receipts from sales of publications at $72,000, of which about $22,000 is expected to be realized from sales of the Book of Standards and its Supplements; miscellaneous receipts, including advertising, interest and dividends are estimated at $15,500. Disbursements have been estimated at $197,650, leaving $1350 for contingencies. The budget provides fully for all the Society's normal operating expenses, including all current publications, activities of standing and district committees, as well as additions to the clerical staff required by the growth of Society activities. Provision has been made for increases in salaries of members of the staff to meet in some measure the present economic conditions; as a result, the salary roll for 1942 on a basis comparable with 1941 has been increased about 10%. The budget also includes the sum of $1000 to be applied to expenses that might need to be incurred in relation to the emergency program on national defense.

The Executive Committee reviews the budget of receipts and disbursements quarterly and makes any adjustments that may be necessary.

The following investments in United States Defense Bonds Series G have been made so far this year: $5000 for the General Account and $3000 for the Research Fund. These investments do not appear in the current auditors' report. They bring the total book value of Society investments as of May 31, 1942, in all funds to $137,982.44; the market value of these securities on the same date was $102,134.73. The Society's cash balance on May 31, 1942, was $97,552.57.

Retirement Plan for Employees:

The Retirement Plan for A.S.T.M. Employees has, as of May 3, 1942, been in effect for four years along the lines set forth in previous reports. Of the 25 employees now on the Society's staff

Report of the Auditors for the Fiscal Year, January 1, to December 1, 1941

Philadelphia, January 7, 1942.

Mr. C. L. Warwick, *Secretary-Treasurer*
American Society for Testing Materials
Philadelphia, Pa.

Dear Sir:

We have examined the books and accounts of your Society for the period from January 1, 1941 to December 31, 1941. We did not make a detailed audit of all transactions, but made extensive tests to determine the accuracy of your records.

We have prepared and submit herewith Balance Sheet as of December 31, 1941, Statement of Cash Receipts and Disbursements for the year ended that date, and other supporting schedules, all of which are in agreement with the records of your Society. In our opinion, these statements present fairly the financial position of the Society at December 31, 1941, and the results of its operations for the year then ended.

The investments owned by the Society, as detailed herein, were examined by us. All income from investments was properly accounted for.

Respectfully submitted,

John Heins & Co.
(Signed) I. Russell Bush
Certified Public Accountant

BALANCE SHEET AS OF DECEMBER 31, 1941

(Including Special and Designated Funds)

Assets

General Funds:			
Current: Cash	$41 961.66		
Less checks drawn as of December 31, 1941—Cost of Proceedings ($12 380.00) and 1941 Supplement to Book of Standards ($11 375.05) in hands of Secretary-Treasurer that date	23 755.05		
		$18 206.61	
Investments (Market Value, $69 267.51)—Cost		88 001.45	
Accounts Receivable		14 120.99	
Total Current Assets—General Fund			$120 329.05
Furniture and Fixtures (depreciated book value)			6 440.58
Total General Funds			$126 769.63
Special and Designated Funds:			
Cash: A.S.T.M. Research Fund		$2 292.11	
Dudley Medal and Marburg Lecture Fund		596.80	
Committee Funds		6 837.97	
			9 726.88
Investments:			
A.S.T.M. Research Fund (Market Value, $21 205.38)—Cost or book value		$29 355.99	
Dudley Medal and Marburg Lecture Fund (Market Value, $2 080.00)—Cost		6 625.00	
Committee Funds (Market Value, $6 394.38)—Cost		6 000.00	
			41 980.99
Total Special and Designated Funds			$51 707.87
Total Assets			$178 477.50

LIABILITIES

General Funds:			
Current: Accounts Payable:			
Committee A-1	$9.74		
Committee C-1	309.79		
Advance Collections	3 617.91		
Total Current Liabilities—General Fund		$3 937.44	
Life Membership Fund	$3 005.20		
Headquarters Fund	5 176.48		
Publication Fund	3 015.00		
Book of Standards Reserve	13 890.25		
Executive Retirement Reserve	1 625.00		
Retirement Fund Reserve	85.75		
Surplus	96 034.51		
		122 832.19	
Total General Funds			$126 769.63
Special and Designated Funds:			
A.S.T.M. Research Fund:			
Principal	$30 391.15		
Income	1 256.95	$31 648.10	
Dudley Medal and Marburg Lecture Fund:			
Principal	6 625.00		
Income	596.80	7 221.80	
Committee Funds		12 837.97	
Total Special and Designated Funds			51 707.87
Total Liabilities and Funds			$178 477.50

COMPARISON OF GENERAL FUNDS FOR FISCAL YEARS 1937–1941, INCLUSIVE

At Close of Fiscal Year	Assets				Liabilities			
	Cash	Investments	Accounts Receivable and Miscellaneous	Furniture and Fixtures	Accounts Payable and Miscellaneous	Reserve for Book of Standards	Miscellaneous Funds	Surplus
1937	$20 686.08	$37 599.49	$10 506.78	$7 029.87	$2 140.86	$6 000.00	$9 061.76	$58 619.60
1938	24 425.51	39 568.24	9 609.00	6 912.96	2 283.88	12 000.00	8 898.99	57 332.84
1939	9 329.65	54 902.10	14 880.53	6 467.27	5 777.01	12 000.00	9 685.40	58 117.14
1940	9 608.61	72 568.95	13 388.88	6 561.93	3 380.25	7 890.25	9 713.79	81 144.08
1941	18 206.61	88 001.45	14 120.99	6 440.58	3 937.44	13 890.25	12 907.43	96 034.51

RECEIPTS AND DISBURSEMENTS

FOR THE PERIOD ENDED DECEMBER 31, 1941

Cash balance, January 1, 1941			$9 608.61
RECEIPTS			
Operating Receipts (Budgeted):			
Dues and Entrance Fees:			
Current Dues	$90 319.49		
Past Dues	855.83		
Advance Dues	2 316.75		
Income, Life Membership Fund	150.00		
Entrance Fees	3 205.00		
Total Dues		$96 847.07	
Sale of Publications:			
Separate Standards	7 393.81		
Book of Standards (Members, additional parts)	5 856.00		
1939 Book of Standards	16 591.16		
1940 Supplement to Book of Standards	10 228.12		
1941 Supplement to Book of Standards	3 892.63		
Methods of Chemical Analysis of Metals	991.99		
Proceedings	3 605.25		
Collective Index to Papers and Reports	53.00		
Selected Standards for Students in Engineering	3 158.27		
Bulletin Subscriptions (Non-Members)	356.00		
Special Reprints and Miscellaneous	27 307.84		
Total Sale of Publications		$79 434.07	
Miscellaneous:			
Binding (Members)	185.00		
Advertising, Bulletin	8 910.89		
Advertising, Index	1 687.50		
Interest and Dividends	4 675.54		
Registration and Other Fees, Annual Meeting	1 846.00		
A.S.T.M. Exhibit, Annual Meeting	4 868.10		
Committee A-1 for Technical Assistant	878.53		
Miscellaneous	167.90		
Total Miscellaneous Items		$23 219.46	
Total Operating Receipts (Budgeted)		$199 500.60	
Nonoperating Receipts (Not Budgeted):			
Investments, Matured or Sold	$3 150.00		
Excess Remittances	149.89		
Accrued Interest	72.89		
Committee C-1 for Technical Assistant	1 800.00		
Executive Retirement Reserve Income	75.00		
Retirement Fund Reserve	6.64		
Total Nonoperating Receipts		$5 254.42	
Total Receipts			$204 755.02
Carried Forward			$214 363.63

Brought forward			$214 363.63

DISBURSEMENTS

Operating Disbursements (Budgeted):			
Publications:			
Year Book	$2 339.12		
1941 Supplements to Book of Standards	17 391.32[a]		
Methods of Chemical Analysis of Metals	41.85		
Proceedings	15 854.12[b]		
Preprints	3 552.76		
Spring Meeting Papers	464.74		
A.S.T.M. Bulletin	8 263.62		
Circulars to Members	1 570.30		
Separate Standards	5 218.19		
Selected Standards for Students	1 715.45		
Index to Standards and Tentative Standards	2 523.24		
Collective Index to Papers and Reports	48.38		
Reprints (Reports, Papers, Symposiums, etc.)	11 506.18		
Miscellaneous Printing (Authors' Reprints, etc.)	1 097.58		
Resetting Standards (double-column)	3 800.00		
Total Disbursements, Publications		$75 386.85	
Salaries		62 152.14	
General Office Expenses		13 989.15	
Expenses, Standing and District Committees		3 369.44	
Expenses, Meetings and A.S.T.M. Exhibit		6 096.38	
Traveling Expenses, Administrative and Special Committees		2 214.26	
American Standards Assn. and Sectional Committees		1 000.00	
Rent, Headquarters		6 250.00	
Furniture and Fixtures		801.02	
Principal, A.S.T.M. Research Fund (one-half current year's entrance fees)		1 600.00	
Employees Retirement Fund		1 471.54	
Emergency National Defense Expense		773.13	
Miscellaneous (Dues, Contributions, etc.)		394.00	
Total Operating Disbursements (Budgeted)		$175 497.91	
Nonoperating Disbursements (Not Budgeted):			
Investments	$18 417.50		
Accrued Interest	103.72		
Refund of Excess Remittances	149.89		
Technical Assistant, Committee C-1	1 600.00		
Headquarters Fund	388.00		
Total Nonoperating Disbursements		$20 659.11	
Total Disbursements			$196 157.02
Cash Balance, December 31, 1941			$18 206.61

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[a] This amount includes $11,375.05 covering checks drawn against cost of 1941 Supplement to the Book of Standards but not actually paid on December 31, 1941.

[b] This amount includes $12,380.00 covering checks drawn against cost of *Proceedings*, but not actually paid on December 31, 1941.

In addition to the general funds of the Society, the Secretary-Treasurer has on hand the following funds as of December 31, 1941:

A.S.T.M. RESEARCH FUND

	Total	*Invested*	*Uninvested Cash*
Principal Account:			
Balance January 1, 1941	$28 791.15	$27 400.99	$1 390.16
Receipts:			
Contribution—A.S.T.M. Current Entrance Fees	+1 600.00		+1 600.00
	$30 391.15	$27 400.99	$2 990.16
Disbursements: Purchase of Securities		+1 955.00	−1 955.00
Balance, December 31, 1941	$30 391.15	$29 355.99	$1 035.16
Income Account:			
Balance January 1, 1941	$1 094.49		
Receipts:			
Interest and Dividends	1 187.48		
Refund, University of Illinois, Speed of Testing (Committee E-1)	293.98		$2 575.95
Disbursements:			
Study to Determine Results of Empirical Flow Tests (Committee E-1) (Brooklyn Polytechnic Inst.)	$500.00		
Soil Tests (Committee D-18) (Dartmouth College)	19.00		
Research Committee on Fatigue of Metals	800.00		$1 319.00
Cash Balance, December 31, 1941			$1 256.95

DUDLEY MEDAL AND MARBURG LECTURE FUND

Balance, January 1, 1941:			
Principal—Investment (at cost)	$6 625.00		
Income—Cash	311.38	$6 936.38	
Receipts			
Interest on Investments		545.62	$7 482.00
Disbursements			
Engrossing 1941 Lecture Certificate		$3.25	
Honorarium, 1941 Lecturer		200.00	
Cost of 1941 Medal		35.00	
Miscellaneous		21.95	260.20
			$7 221.80
Balance, December 31, 1941:			
Principal—Investment (at cost)		$6 625.00	
Income—Cash		596.80	$7 221.80

COMMITTEE FUNDS

Committee A-1 on Steel	$745.17	
Committee A-5 on Corrosion of Iron and Steel	3 050.60	
Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys	2 951.11	
Committee B-6 on Die-Cast Metals and Alloys	52.10	
Committee C-1 on Cement	838.91	
Committee C-1—Cement Reference Laboratory	939.83	
Committee C-9 on Concrete and Concrete Aggregates	192.52	
Committee C-9—Sanford E. Thompson Medal Fund	466.65	
Committee C-15 on Manufactured Masonry Units	331.36	
Committee C-18 on Natural Building Stones and Slate	170.36	
Committee D-1 on Paint, Varnish, Lacquer and Related Products	80.00	
Committee D-2 on Petroleum Products and Lubricants	95.04	
Committee D-5 on Coal and Coke	1 235.70	
Committee D-13 on Textile Materials	409.02	
Committee D-13—Testing Textile Finishes	304.37	
A.S.A. Sectional Committee on Specifications for Cast-Iron Pipe and Special Castings	138.18	
Research Committee on Fatigue of Metals	837.05	
Total Committee Funds		$12 837.97
Accounted for as follows:		
Cash Balance, December 31, 1941	$6 837.97	
Investments—at cost	6 000.00	
		$12 837.97

AMERICAN SOCIETY FOR TESTING MATERIALS INVESTMENTS (AS OF DECEMBER 31, 1941)

Number of Shares or Par Value		*Cost or Book Value*	*Market Value*
	GENERAL FUNDS		
	Bonds		
$3000	Armour & Co., of Del., First Mortgage, 20-yr., Sinking Fund, Series C, 4's, due 1957	$2 955.00	$3 123.75
9000	Balto. & Ohio R. R., Refunding and General Mortgage 5's, Series F, due 1996	9 106.55	2 891.25
2000	Columbia Gas & Electric Co., Debentures, 5's, due 1961	2 025.34	1 990.00
500	Florida Power & Light Co., First Mortgage, 5's, due 1954	491.25	515.00
4000	General Steel Castings Corp., First Mortgage, Series A, 5½'s, due 1949	3 610.00	3 880.00
3000	Michigan Consolidated Gas Co., First Mortgage, 4's, due 1963	2 925.00	3 157.50
2000	North Station Industrial Bldg., Inc., First Mortgage, Sinking Fund, 5's, due 1962[a]	1 850.00	1 600.00
4000	N. Y., Chicago & St. Louis R. R., Refunding Mortgage, Series C, 4½'s, due 1978	2 805.61	2 510.00
3000	Pressed Steel Car Co., Inc., 15-yr. Debentures, 5's, due 1951	2 868.75	2 700.00
2000	U. S. Defense Savings Bond, Series G, due July 1, 1953[b]	2 000.00	2 000.00
3800	U. S. Treasury Bonds, 2½'s, due 1967–72	3 800.00	3 800.00
		$34 437.50	$28 167.50
	Stock		
30	Alvin Progressive Federal Savings & Loan Assn.[b]	$3 000.00	$3 000.00
25	American Can Co., common	2 122.30	1 500.00
13	American Cyanamid Co., 5 per cent Cumulative preference	130.00	151.13
50	American Cyanamid Co., Class B common	1 764.25	2 037.50
100	American Radiator & Std. Sanitary Corp., common	1 564.32	412.50
25	American Tobacco Co., common	2 110.12	1 131.25
100	Atlantic Refining Co., common, par value $25	2 302.50	2 275.00
100	Borden Co.	1 989.00	1 912.50
25	Colgate-Palmolive-Peet Co., $4.25 preferred	2 525.00	2 543.75
30	Collingdale Federal Savings & Loan Assn.[b]	3 000.00	3 000.00
50	Gen. Elec. Co. common, no par value	2 006.25	1 331.25
50	General Telephone Corp. $2.50 preferred[a]	2 675.00	2 375.00
50	Great Northern Ry. $6 preferred par value $100	1 774.83	1 062.50
25	Hazel Atlas Glass Co.	2 510.64	2 275.00
100	S. S. Kresge Co., common	2 490.18	2 112.50
100	Mathieson Alkali Works, common, no par value	3 016.00	2 725.00
25	J. C. Penney Co., common	2 279.63	1 937.50
100	Public Service Corp. of N. J., common	4 129.50	1 237.50
25	Sears, Roebuck & Co., Capital	1 760.71	1 303.13
200	United Gas Improvement Co., common	2 578.00	925.00
50	U. S. Gypsum	3 819.27	2 150.00
40	Westinghouse Elec. & Mfg. Co., common	3 456.45	3 085.00
8	Westinghouse Elec. & Mfg. Co. (dividend shares)	560.00	617.00
		$53 563.95	$41 100.01
		$88 001.45	$69 267.51

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[a] Market values were taken from current financial publications with the exception of quotations on inactive or unlisted securities indicated by footnote[a]. These values were obtained from brokers as of December 31, 1941.

[b] In the absence of a quoted market value, these securities are included at their cost which is equal to their face value.

	A.S.T.M. RESEARCH FUND		
	Bonds		
$2000	First Mortgage Real Estate, 1315–1317 Spruce St., 6's, due 1945[a]	$2 000.00	$700.00
1000	Allis Chalmers Mfg. Co., Convertible Debentures, 4's, due 1952	1 035.28	1 077.50
1500	Balto. & Ohio R. R., General & Refunding, 5's, Series F, due 1996	1 511.25	481.88
2000	Columbia Gas & Electric Co., Debentures, 5's, due 1961	2 025.31	1 990.00
1500	Florida Power & Light Co., First Mortgage, 5's, due 1954	1 473.75	1 545.00
3000	North Station Industrial Bldg. Inc., First Mortgage, Sinking Fund, 5's, due 1962[a]	2 812.50	2 400.00
2000	N. Y., Chicago & St. Louis, Refunding & Mortgage, Series C, $4\frac{1}{2}$'s, due 1978	1 400.26	1 255.00
3000	Republic Steel Corp., General Mortgage, Series B, $4\frac{1}{2}$'s, due 1961	3 000.00	3 105.00
		$15 258.35	$12 554.38
	Stock		
12	Consolidated Edison Co. of N. Y. $5 preferred	$1 234.39	$1 053.00
29	Consumers Power Co. $4.50 preferred, no par value	2 914.50	2 697.00
98	General Motors Corp., common, par $10[c]	6 504.75	3 013.50
25	Pittsburgh Plate Glass Co., common	1 955.00	1 425.00
100	United Gas Improvement Co., common	1 489.00	462.50
		$14 097.64	$8 651.00
		$29 355.99	$21 205.38
	DUDLEY MEDAL & MARBURG LECTURE FUND		
$500	Balto. & Ohio R. R., Refunding & General Mortgage 6's, Series C, due 1995	$550.00	$175.00
6000	Balto. & Ohio R. R., Refunding & General Mortgage 5's, Series A, due 1995	6 075.00	1 905.00
		$6 625.00	$2 080.00
	COMMITTEE FUNDS		
$5000	U. S. Treasury Bonds, $2\frac{3}{4}$'s, due 1947—45	$5 000.00	$5 300.00
1000	U. S. Treasury Bonds, $2\frac{7}{8}$'s, due 1960—55	1 000.00	1 094.38
		$6 000.00	$6 394.38
	Grand Total	$129 982.44	$98 947.27

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[c] The income from these shares is, by agreement, paid to an annuitant.

three are not yet eligible for participation in the plan, and of the remainder, 19 are participating. With the exception of some of the younger members of the staff all the participants are contributing $2\frac{1}{2}$ per cent or more of their salaries, and the Society's annual contribution at the present rate of about $1680 is slightly under $2\frac{1}{2}$ per cent of the salary roll. The Executive Committee would like to see this percentage increased to provide better retirement incomes; but in view of the uncertainty of the times and the possibility that the Social Security Act may be amended to include within its scope such organizations as A.S.T.M., it has been decided to maintain the Retirement Plan at its present level, with the understanding that new employees will be admitted into the Plan as they become eligible and that additional contracts may be arranged in some instances as a recognition of years of service and to provide for a more equitable retirement income in relation to salary.

Cooperative Activities:

The Society's cooperative relations with other organizations have continued along well-established lines. News items in the ASTM BULLETIN have told of various activities in this direction and these need not be repeated. Several happenings during the year, however, merit specific mention here.

The Society's contacts with the American Standards Association constitutes one of its most important cooperative activities. The principal work has been more or less routine in nature, involving the submission of new and revised standards to the A.S.A. for approval as American standards, which is presented in detail in the annual report of Committee E-10 on Standards.

The Society has assumed joint sponsorship with the American Association of Textile Chemists and Colorists for a new A.S.A. Sectional Committee on Fastness of Color Textiles and has designated W. M. Scott and A. G. Ashcroft as its representatives, with the Assistant Secretary as alternate. It has also accepted an invitation to be represented on a new A.S.A. Sectional Committee on Building Code Requirements for Fire Protection and Fire Resistance, on which H. M. Robinson has been designated as representative with G. N. Thompson as alternate. R. P. Miller has been re-appointed for a term of two years as the Society's representative on the Building Code Correlating Committee. F. S. Mapes has been appointed to represent Committee D-13 on the Sectional Committee on Electrical Insulating Materials and R. M. Havourd has been designated to represent Committee D-9 on Sectional Committee on Glass Insulators, to replace Merrill de Merit, resigned.

The Society has continued to maintain contact through the American Standards Association with the subject of engineering relations between the American Republics, to which quite a little attention was devoted in the report to the Society a year ago. Our entry into the war has, of course, profoundly affected the political and economic relations of our country with the South American Republics, and while accentuating the importance of collaboration between the American Republics in the matter of specifications and tests for materials it has at the same time interfered with discussions and development of plans that were under consideration a year ago. However, the Office of Inter-American Affairs has recently expressed considerable interest in the representations that have been made to it by the A.S.A. for establishing some suitable means of promoting interchange of information on standards and discussions are currently being held that may lead to important actions.

During the year the Society joined with the National Research Council in sponsoring a Joint Committee on Chemical Analysis by X-ray Diffraction Methods, under the chairmanship of W. P. Davey of The Pennsylvania State College. Since that time the National Research Council decided to discontinue its Committee on X-ray and Electron Diffraction and to transfer its activities to the American Society for X-ray and Electron Diffraction, and this latter society is now joint sponsor with the A.S.T.M. for this project. The A.S.T.M. has handled for the Joint Committee the publication and sale of cards giving X-ray diffraction data for identification of chemical compounds and it is expected that part of the profits from the sales of these cards will be turned over to the Joint Committee to support its development work.

Respectfully submitted on behalf of the Executive Committee,

G. E. F. LUNDELL,
President.

C. L. WARWICK,
Secretary-Treasurer.

June, 1942.

REPORT OF COMMITTEE A-1

ON

STEEL

Two situations have affected markedly the activities of Committee A-1 on Steel during the past year—first, the National Emergency created by the war; secondly, the fact that the year 1942 is the A.S.T.M. Book of Standards' year.

The war situation has led to several actions by the committee and its members, some of which are discussed below, including the National Emergency Steel Specifications' activities and the development of emergency alternate provisions.

Preceding the publication of the triennial Book of Standards, Committee A-1 on Steel, like all other standing committees, is endeavoring to bring its specifications into the best possible shape for publication in the book. During the past year this has involved a review of the some 120 specifications for which it is responsible. As a result, a number of actions are being taken—Table I providing an analysis of the letter ballot vote, and indicating those tentative specifications and standards on which some action is proposed. The report also lists, with comment, emergency alternate provisions which have been issued, and discusses four tentative standards issued in April.

Meetings.—Three series of meetings have been held during the year: the first in Chicago, Ill., on June 24 and 25, 1941; the second in Philadelphia, Pa., on January 12 and 13, 1942; and the third in Cleveland, Ohio, on March 2 and 3, 1942. Including subcommittee and section meetings, the total number of meetings held during the year has exceeded 45. Despite the large number of other meetings which members of Committee A-1 have had to attend, including those of the various committees appointed as a part of National Emergency Steel Specifications project, there has been no diminution in the attendance at the A-1 sessions which is indicative of the continued close cooperation of a large percentage of the committee personnel and the importance with which the work is viewed by the various representatives. A definite effort has been made during the year to conserve the members' time by handling many matters through correspondence.

Personnel.—Since the 1941 annual meeting, 20 new members have been elected and 5 resignations accepted, the committee membership now totaling 230, of whom 120 are classified as nonproducers and 110 as producers; of the former, 90 are consumers and 30 are general interest members. In addition to the regular members, the committee has had the close cooperation of a number of consulting members who have served in connection with several subcommittee activities, notably in the development of specifications for pipe piles and the end-quench test for hardenability.

Losses by Death.—During the year Committee A-1 has lost by death five members: H. B. Bent, Bethlehem Steel Co.; J. C. Craven, Federal Shipbuilding and Dry Dock Co.; G. L.

Norris, Vanadium Corporation of America; Harold J. Stein, Allis-Chalmers Mfg. Co.; and W. E. Woodard, Lima Locomotive Works, Inc. The committee records its appreciation of their interest and services.

NATIONAL EMERGENCY STEEL SPECIFICATIONS

Although Committee A-1 on Steel is not officially a part of the NESS project (announcements of which have appeared from time to time in the ASTM BULLETIN) it seems appropriate in this report to record its development because cooperating closely in the work are a great many members of Committee A-1, representing both consumers and producers. Further, the work is bound to influence to some extent the committee's standardization activities. This project which has as its aim the increased production of steel by concentration on a minimum number of compositions, specifications, etc., was launched by the former Office of Production Management with three national organizations active in this field carrying out the work, namely, the Society of Automotive Engineers, the American Iron and Steel Institute, and the American Society for Testing Materials. Closely cooperating in the work are the War and Navy Depts. At the request of Donald M. Nelson, then Director of Purchases, the Society's Secretary-Treasurer, C. L. Warwick, who was then serving as Consultant on Specifications in the Office of Production Management, was named Administrator and is Chairman of the Administrative Committee, which consists of representatives from the five bodies named. A statement of the objective and the supplementary statement are as follows:

> To establish as promptly as possible a selected list of steel specifications, to be designated as List of National Emergency Steel Specifications, which in effect involves the selection from specifications now promulgated of the minimum number of steel specifications, compositions, and sections necessary to meet the requirements of National Defense, both direct and indirect.
>
> It is believed that the productive capacity of the steel industry and of the manufacturing industries using steel for defense requirements can be materially increased within present facilities by concentration of production upon a reduced number of steels, particularly with respect to alloy steels.
>
> It is understood that consideration must necessarily be given to civilian requirements for steel in establishing the List of National Emergency Steel Specifications.
>
> It is the intention of OPM, through its Iron and Steel Section, to use the List of National Emergency Steel Specifications as an aid in administering steel priorities and allocations.

The representative of the Society on the Administrative Committee is N. L. Mochel, Manager, Metallurgical Engineering, Westinghouse Electric and Manufacturing Co., and Chairman of Committee A-1 on Steel, with Jerome Strauss, Vice-President, Vanadium Corporation of America and Chairman of Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys as alternate. R. J. Painter, Staff Assistant, Committee A-1, has devoted considerable time assisting Mr. Mochel and in his capacity as Assistant to the Secretary A.S.T.M., has aided Mr. Warwick in various phases of the work.

In selecting personnel representing the consuming interests and the producing interests, it would be expected that many Committee A-1 members who for years have been active in the development of standard specifications for steel would be asked to serve on the various Technical Advisory Committees. Personnels of these groups have been published in the October and December, 1941, ASTM BULLETINS, and the January and March, 1942, issues. Too much credit cannot be accorded these men for their services during this period when their other interests are demanding more and more of their time.

A list of the Technical Advisory Committees, which have been organized, with their chairmen, follows:

- TAC 1 on Carbon Steel Plates—Oscar U. Cook, Asst. Mgr., Dept. of Metallurgy, Inspection & Research, Tennessee Coal, Iron & Railroad Co.
- TAC 2 on Alloy Steel Plates—E. F. Kenney, Metallurgical Engineer, Bethlehem Steel Co.
- TAC 3 on Aeronautical Steels—J. B. Johnson, Chief, Materiel Laboratory, Air Corps, U. S. A.
- TAC 4 on Structural Steel Shapes—F. H. Frankland, Director of Engineering, American Institute of Steel Construction, Inc.
- TAC 5 on Rails and Track Accessories—H. H. Morgan, Chief Engineer, Robert W. Hunt Co.
- TAC 6 on Wrought Steel Wheels—C. T. Ripley, Chief Engineer, Technical Board, Wrought Steel Wheel Industry.
- TAC 7 on Carbon and Alloy Steel Bars—John Mitchell, Metallurgical Engr., Alloy, Carnegie-Illinois Steel Corp.
- TAC 8 on Concrete Reinforcement Steel—H. P. Bigler, Assistant to the President, Connors Steel Co.
- TAC 9 on Rolled or Forged Axles and Railroad Forgings—John D.Tyson, Chief Metallurgist, Standard Steel Works Division, Baldwin Locomotive Works.
- TAC 11 on Tubular Steel Products—T. G. Stitt, Chief Inspecting Engineer, Pittsburgh Steel Co.
- TAC 16 on Heavy Forgings—C. J. Boyle, Works Laboratory, General Electric Co.

While no definite orders have yet been issued by the War Production Board, a number of immediate benefits and advantages of the work were obvious from the beginning because those participating, extremely influential metallurgists and technologists, carried from the meetings a better conception of some of the problems involved and put into practice a number of the matters discussed. Consideration of specifications issued by various bodies, including a large number of A.S.T.M. specifications, directed attention to modifications which should be made, primarily of an emergency matter but some of which undoubtedly will remain effective after the war. A number of the emergency alternate provisions recommended by Committee A-1 developed as a result of this NESS work.

CONSERVATION OF CRITICAL OR STRATEGIC MATERIALS

Greatly increased production of steel, and particularly alloy steel, considered so essential for many ordnance and related uses, and the possibility of greatly reduced importations of certain alloying elements, such as manganese and chromium, necessitates the conserving of these alloying elements wherever possible so that the stocks we have can be used most effectively.

To determine what emergency changes might be made in specifications to conserve these elements, in particular, manganese, studies were made by the various subcommittees of Committee A-1. Although some actions have been taken as recommended by Subcommittee I on Steel Rails and Accessories, Subcommittee IV on Spring Steel and Steel Springs, and Subcommittee VIII on Steel Castings, each of which makes a definite contribution, it is probable that some further actions will be necessary.

In general the specifications under the jurisdiction of Committee A-1 are of three types as regards the controlling factors: first, where chemical compositions are the chief governing requirements; second, where chemical and physical requirements are both specified; and third, where physical requirements are predominant, possibly with phosphorus and sulfur limitations as the only chemical limitations. Most of the specifications fall in the latter two groups with many in the last-named classification, which throws the burden of conservation so far as elements entering into the composition are concerned on producers. There is no question that every possible step is being taken by them to conserve these alloying elements.

In certain fields qualified technical committees have recommended marked emergency increases in the allowable design stresses. These actions will conserve steel; they have a very distinct bearing on any consideration which may be given to the reduction in physical requirements in specifications.

GLOSSARY OF HEAT TREATING TERMS

On the recommendation of A.S.T.M. Committee E-8 on Nomenclature and Definitions,[1] there is being reported to the Society for publication as tentative Proposed Definitions of Terms Relating to Heat Treatment of Metals as developed by a joint committee consisting of representatives of the American Society for Metals, Society of Automotive Engineers, the American Foundrymen's Association, and the American Society for Testing Materials. Growing criticism of the previously published terms and the desirability of having a complete meeting of the minds led to the revival of the Joint Committee on Definitions of Terms Relating to Heat Treatment on which A.S.T.M. has three representatives, including Lawford H. Fry who is serving in a dual capacity as the chairman of the Committee E-8 subgroup concerned and of Subcommittee XXIV on Heat Treatment of Steel of Committee A-1. The definitions have been referred informally to the members of Subcommittee XXIV and following approval by the Society will be officially referred to Subcommittee XXIV for comment.

TENTATIVE STANDARDS AND REVISIONS APPROVED DURING THE PAST YEAR

On the recommendation of Committee A-1, six tentative standards were approved[2] during the year by Committee E-10 on Standards, the titles and date of approval being as follows:

Tentative Specifications for:

Atomic-Hydrogen-Arc-Welded and Electric-Resistance-Welded Alloy-Steel Boiler and Superheater Tubes (A 249 - 41 T) (Approved August 25, 1941),

Electric-Resistance-Welded Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 250 - 41 T) (Approved August 25, 1941),

Iron and Steel Arc-Welding Electrodes (A 233 - 42 T)[3] which is an extensive revision of Specications A 233 - 40 T (Approved April 28, 1942), and

Iron and Steel Gas Welding Rods (A 251 - 42 T)[3] (Approved April 28, 1942),

Welded and Seamless Steel Pipe Piles (A 252 - 42 T)[3] (Approved April 28, 1942),

Tentative Method for:

End-Quench Test for Hardenability of Steel (A 255 - 42 T)[3] (Approved April 28, 1942).

Welded Boiler and Superheater Tubes.—Specifications A 249, recognize the two designated processes for welding austenitic steels and cover material which has already been standardized by another process in an A.S.T.M. standard. This same situation holds with respect to Specifications A 250 which extend the range of boiler and pressure vessel tubing and pipe fabricated by electric-resistance-welding, as covered in A.S.T.M. specifications.

Arc-Welding Electrodes and Gas Welding Rods.—In its work on so-called filler metal specifications, Subcommittee XXI on Steel Welding Electrodes and Welding Rods has cooperated very closely with the Filler Metal Specifications Committee of the American Welding Society, each group being headed by J. H.

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[1] See p. 482.

[2] In submitting these recommendations to Committee E-10 on Standards, Committee A-1 reported the following results of the letter ballot vote of a total 157 ballots returned from a committee membership of 231: A 249 - 41 T, affirmative 69, negative 1, ballots marked "not voting" 87; A 250 - 41 T, affirmative 71, negative 0, ballots marked "not voting" 86, and the following results of a total 128 ballots returned from a committee membership of 230: A 233 - 42 T, affirmative 82, negative 0, ballots marked "not voting" 46; A 251 - 42 T, affirmative 75, negative 0, ballots marked "not voting" 53; A 252 - 42 T, affirmative 72, negative 0, ballots marked "not voting" 56; and A 255 - 42 T, affirmative 89, negative 0, ballots marked "not voting" 39.

[3] 1942 Book of A.S.T.M. Standards, Part I.

Deppeler, with a number of men serving on both committees. The new arc welding specifications being issued in complete form are essentially an extended revision of Specifications A 233 – 40 T. A number of important modifications have been made including the introduction of yield strengths for the classes E 60 and E 70 and the addition of all the mechanical data for classes E 80, E 90, and E 100. In addition to this there are included root bend and face bend tests to determine that the electrodes can make suitable joints.

The Tentative Specifications for Iron and Steel Gas Welding Rods (A 251 – 42 T) are suitable for welding carbon and low-alloy steels. The rods are classified on the basis of tensile strength of the deposited weld metal in the stress-relieved condition.

Welded and Seamless Steel Pipe Piles.—After detailed discussion at a meeting of a special section of Subcommittee IX headed by L. H. Winkler, which included a number of design engineers and users of the material, minor substance changes and a number of editorial modifications were incorporated in the original draft. The requirements cover black, furnace welded, electric welded and seamless steel pipe piles and apply only to piles in which the steel tubular section acts as a permanent load-carrying member.

A most important part of the standard is a Table of Standard Weights and Dimensions of Welded and Seamless Steel Pipe Piles.

End-Quench Test for Hardenability of Steel.—There has been much discussion for several years on methods for evaluating the hardenability of steel. The literature has included a number of extensive reports and technical papers and there has been discussion at various meetings, including those of the Society of Automotive Engineers, a group which has been particularly concerned with the test. The desirability of a recognized standard has become increasingly apparent with the critical situation involving use of alternate steels. In October, 1941, the A.S.T.M.'s Executive Committee received a suggestion from a group which had been studying the so-called Jominy hardenability test that it would be extremely helpful to industry to have an A.S.T.M. standard method issued.

The matter was referred to Committee A-1 on Steel which appointed a Subcommittee on Hardenability headed by E. W. Upham, Chrysler Corp. This company had done extensive work in this field with a group which included in addition to its own metallurgists many technical men from various steel companies. The committee concluded that the arithmetic method of plotting was most desirable at this stage and accordingly recommended it. The arithmetic chart is the one used in the Iron and Steel Institute Manual No. 5 on Possible Alternates for Nickel, Chromium and Chromium-Nickel Constructional Alloy Steels. It differs from the logarithmic chart incorporated in the methods developed in an S.A.E. subcommittee.

Hardenability is measured quantitatively usually by noting the extent or depth of hardening of a standard size and shape of test specimen in a standardized quench. In the end-quench test the "depth of hardening" is the distance along the specimen from the quenched end for a given degree of hardening.

EMERGENCY STANDARD FOR BALL AND ROLLER BEARING STEELS

This important new emergency standard for ball and ball bearing steel (ES – 5) which resulted from the work of the NESS has been officially referred to Committee A-1 for such action as it might wish to take regarding further revisions or acceptance as an A.S.T.M.

tentative standard. One of the most important NESS Technical Advisory Committees covers Carbon and Alloy Steel Bars, Blooms, and Billets. It has several subcommittees, one of which concerns bearing steels headed by L. A. Lanning, New Departure Division, General Motors Corp., the subcommittee personnel including leading metallurgists and technical men representing industrial consumers and general interests from the roller bearing and ball bearing fields, and steel producers. Since there was no generally applicable specification the committee devoted considerable time in cooperation with the Iron and Steel Branch, War Production Board, in drafting a standard.

The personnel of the Bearing Steel Section was organized basically along the same lines as an A.S.T.M. group, that is, of producers, consumers, and general interests, and the S.A.E. and the A.I.S.I., co-sponsors with A.S.T.M. of the NESS project, agreed that the A.S.T.M. was the logical one of the sponsors to promulgate the specification. The A.S.T.M. Executive Committee at its April meeting received the specifications and referred them directly to Committee E-10 on Standards with the recommendation that they be approved on the basis of the manner in which they had been developed and the considerations that had entered into their development. If approved, the specifications were then to be referred to Committee A-1. As these specifications were approved by Committee E-10 for publication as an Emergency Standard on April 28, 1942, they will be discussed at the next meeting of Committee A-1 with the probability that a special subcommittee will be formed, including many of those active in preparing the specifications.[4]

EMERGENCY ALTERNATE PROVISIONS

Committee A-1 has made extensive use of the procedure established by the Society to effect emergency alternate provisions.

A list of the Emergency Alternate Provisions, with comments on certain of them, follows:

Designation		Issued
	Standard Specifications for:	
EA - A 1	Open-Hearth Carbon-Steel Rails (A 1 - 39)	4.6.42
EA - A 26	Steel Tires (A 26 - 39)	4.28.42
EA - A 27	Carbon-Steel Castings for Miscellaneous Industrial Uses (A 27 - 39)	2.24.42
EA - A 67	Steel Tie Plates (A 67 - 33)	4.6.42
EA - A 87	Carbon-Steel and Alloy-Steel Castings for Railroads (A 87 - 36)	2.24.42
EA - A 148	Alloy-Steel Castings for Structural Purposes (A 148 - 36)	2.24.42
EA - A 160	Axle-Steel Bars for Concrete Reinforcement (A 160 - 39)	4.6.42
EA - A 194	Carbon and Alloy-Steel Nuts for Bolts for High-Pressure and High-Temperature Service to 1100 F. (A 194 - 40)	4.6.42
EA - A 215	Carbon-Steel Castings Suitable for Fusion Welding for Miscellaneous Industrial Uses (A 215 - 41)	2.24.42
EA - A 227	Hard-Drawn Steel Spring Wire (A 227 - 41)	4.6.42
EA - A 229	Oil-Tempered Steel Spring Wire (A 229 - 41)	4.6.42
EA - A 230	Carbon-Steel Valve Spring Quality Wire (A 230 - 41)	4.6.42
EA - A 241	Hot-Worked High-Carbon Steel Tie Plates (A 241 - 41)	4.6.42
	Tentative Specifications for:	
EA - A 183	Heat-Treated Carbon- and Alloy-Steel Track Bolts and Nuts (A 183 - 40 T)	4.6.42
EA - A 216	Carbon-Steel Castings Suitable for Fusion Welding for Service at Temperatures up to 850 F. (A 216 - 41 T)	4.6.42
EA - A 217	Alloy-Steel Castings Suitable for Fusion Welding for Service at Temperatures from 750 to 1100 F. (A 217 - 41 T)	4.6.42

The complete provisions have appeared in the A.S.T.M. BULLETIN and had been sent as "pink slips" to all members of the committee and are being distributed to members of the Society. Additional copies can be obtained from A.S.T.M. Headquarters on request.

Rails and Track Accessories.—These provisions resulted from discussion in the National Emergency Steel Technical Advisory Committee 5 and were effected by Subcommittee I at the same time that the American Railway Engineering Association was taking similar action. The provisions for tie plates, by eliminating references to copper, tend to conserve this element and the deletion of references to alloy-steel track bolts in Specifications A 183 and a modification of the requirement on wrench fit tightness of nuts are in line with the basic aim of these emergency provisions. The several provisions in the rail specifications also will tend to help in procurement by changes in the sections covering chemical composition, drop test, inclusion of requirements on progressive all-ingot nick and break tests, deletion of the Brinell hardness requirement; while the inclusion of recommended practices for controlled cooling adds desirable information in the specifications.

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[4] A special subcommittee has been organized under the chairmanship of J. J. Lanning and on its recommendation revisions in the emergency specifications became effective September 16, 1942, with the designation ES-5a, see Editorial Note, p. 94.

Spring Wire.—Detailed review by Subcommittee IV on Spring Steel and Steel Springs of all of its specifications resulted in provisions deleting as an emergency matter the manganese requirements in the specifications for spring wire listed above. No change was made in the physical requirements, which will still insure the quality of the material to the purchaser. In other specifications the committee felt that no emergency requirements would effect, safely at least, any conservation in alloying elements.

Steel Tires.—This modification effected in the specifications for steel tires on the recommendation of Subcommittee VII on Steel Wheels and Steel Tires raises the permissible sulfur for acid steel from 0.05 and 0.06 per cent maximum, and eliminates the requirement on residual nickel and chromium.

Steel Castings.—The emergency provisions in the steel casting specifications will bring certain of the requirements in line with other specifications, particularly Federal Specification QQ-S-681b and proposed Navy requirements, and should be of definite help in production.

The omission of the manganese requirements from Specifications A 27 would permit a foundry to furnish steel from a low-alloy heat instead of making a special higher carbon steel. The change in physical properties of grade B2 in Specifications A 27 will permit supply from heats of certain classes of Federal and Navy castings. There is no change in the yield point requirement important in design.

The change in Specifications A 87 concerning weight of casting for which test coupons must be provided brings the requirement in line with other A.S.T.M. specifications and definitely expedites production.

The emergency grade 4, of Class 4, provided for in Specifications A 148 is similar to a widely used class in Federal specifications and should be covered by A.S.T.M.

The provisions in Specifications A 215 reduce the number of grades of weldable carbon steel from 8 to 5 and permit the pouring of castings from heats which might also be used for Federal or proposed Navy specifications. Here again the yield point requirement is changed only to a minor extent.

The emergency provisions in Specifications A 215 and A 216 cover a use of material for pressure containing turbine castings which have been specified in accordance with these two standards, but which have really not been applicable until the emergency changes now cover this use.

Axle-Steel Reinforcing Bars.—This modification provides for a much wider source of supply than in the present standard. The proposed addition of a new emergency grade of soft structural steel has met with considerable objection and this recommendation is being held in abeyance.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Spec. for Welded Alloyed Open-Hearth Iron Pipe (A 253 – 42 T)	92	0	60
Spec. for Copper Brazed Steel Tubing (A 254 – 42 T)	75	0	77
Spec. for Lap-Welded and Seamless Steel Pipe for High-Temperature Service (A 106 – 42 T)—reverted to status of tentative standard, and revised	100	1	51
II. REVISIONS OF TENTATIVE STANDARDS			
Spec. for Light Gage Structural Quality Flat Hot-Rolled Carbon Steel (0.2499 and 0.1874 in. to 0.0478 in. in Thickness) (A 245 – 41 T)	81	3	75
Spec. for Ligh Gage Structural Quality Flat Rolled Carbon Steel (0.0477 to 0.0225 in. in Thickness) (A 246 – 41 T)	85	0	74
Spec. for Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F. (A 158 – 41 T)	85	0	74
Spec. for Seamless Carbon-Molybdenum Alloy-Steel Pipe for Service at Temperatures from 7500 to 1000 F. (A 206 – 41 T)	85	0	74
Spec. for Carbon-Steel Castings Suitable for Fusion Welding for Service at Temperatures up to 850 F. (A 216 – 41 T)	111	0	41
Spec. for Alloy-Steel Castings Suitable for Fusion Welding for Service at Temperatures from 750 to 1100 F. (A 217 – 41 T)	107	0	45
III. REVISIONS OF STANDARDS, IMMEDIATE ADOPTION			
Spec. for Chromium-Vanadium Steel Bars for Springs (A 60 – 39)	87	0	72
Spec. for Seamless Cold-Drawn Low-Carbon Steel Heat-Exchanger and Condenser Tubes (A 179 – 40)	88	1	70
Spec. for Electric-Resistance-Welded Steel Heat-Exchanger and Condenser Tubes (A 214 – 40)	88	1	70
Spec. for Seamless Alloy-Steel Boiler and Superheater Tubes (A 213 – 40)	91	1	67
Spec. for Welded and Seamless Steel Pipe (A 53 – 40)	98	2	53
Spec. for Black and Hot-Dipped Zinc-Coated (Galvanized) Welded and Seamless Steel Pipe for Ordinary Uses (A 120 – 40)	93	3	63
Spec. for Molybdenum-Steel Plates for Boilers and Other Pressure Vessels (A 204 – 39)	93	0	66
Spec. for Carbon-Steel Plates for Stationary Boilers and Other Pressure Vessels (A 70 – 39)	94	0	65
Spec. for Low-Carbon Nickel-Steel Plates for Boilers and Other Pressure Vessels (A 203 – 39)	91	0	68
Spec. for Commercial Quality Hot-Rolled Bar Steels (A 107 – 40)	96	0	63
Spec. for Alloy-Steel Castings for Valves, Flanges, and Fittings for Service at Temperatures from 750 to 1100 F. (A 157 – 41)	89	0	70
Spec. for Carbon-Steel Bars for Springs (A 14 – 39)	83	1	68
IV. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Spec. for Low-Alloy Structural Steel (A 242 – 41 T)	99	0	60
Spec. for Carbon-Steel Forgings for General Industrial Use (A 235 – 40 T)	107	0	52
Spec. for Carbon-Steel Forgings for Locomotives and Cars (A 236 – 40 T)	98	0	61
Spec. for Alloy-Steel Forgings for General Industrial Use (A 237 – 40 T)	106	0	53
Spec. for Alloy-Steel Forgings for Locomotives and Cars (A 238 – 41 T)	98	0	61
Spec. for Factory-Made Wrought Carbon-Steel and Carbon-Molybdenum-Steel Welding Fittings (A 234 – 40 T)	89	0	70
Spec. for Atomic-Hydrogen-Arc-Welded and Electric-Resistance-Welded Alloy-Steel Boiler and Superheater Tubes (A 249 – 41 T), as revised	79	0	73
Spec. for Seamless Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 209 – 41 T)	85	0	67
Spec. for Heat-Treated Wrought Steel Wheels (A 244 – 41 T)	75	0	77
V. ADOPTION AS STANDARD OF TENTATIVE REVISIONS OF STANDARDS			
Spec. for Steel for Bridges and Buildings (A 7 – 39)	92	0	67
Spec. for Structural Steel for Locomotives and Cars (A 113 – 39)	92	0	67
Spec. for Carbon-Steel Castings for Miscellaneous Industrial Uses (A 27 – 39)	101	0	58
Spec. for Carbon-Steel and Alloy-Steel Castings for Railroads (A 87 – 36)	101	0	58
Spec. for Alloy-Steel Castings for Structural Purposes (A 148 – 36)	101	0	58
Spec. for Welded and Seamless Steel Pipe (A 53 – 40)	92	0	67
Spec. for Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes (A 83 – 40)	92	0	67
Spec. for Black and Hot-Dipped Zinc-Coated (Galvanized) Welded and Seamless Steel Pipe for Ordinary Uses (A 120 – 40)	92	0	67
Spec. for Electric-Fusion-Welded Steel Pipe (Sizes 30 in. and Over) (A 134 – 39)	90	0	69
Spec. for Electric-Resistance-Welded Steel Pipe (A 135 – 34)	90	0	69
Spec. for Electric-Fusion-Welded Steel Pipe (Sizes 8 in. to but not Including 30 in.) (A 139 – 39)	89	0	70
Spec. for Seamless Alloy-Steel Boiler and Superheater Tubes (A 213 – 40)	92	0	67
Spec. for Forged or Rolled Steel Pipe Flanges for General Service (A 181 – 37)	92	0	67
Spec. for Seamless Cold-Drawn Alloy-Steel (4 to 6 per cent Chromium) Heat-Exchanger and Condenser Tubes (A 187 – 40)	92	0	67
Spec. for Boiler and Firebox Steel for Locomotives (A 30 – 39)	91	3	65
Spec. for Electric-Fusion-Welded Steel Pipe for High-Temperature and High-Pressure Service (A 155 – 36)	88	0	71

TABLE I.—*Concluded.*

Items	Affirmative	Negative	Ballots Marked "Not Voting"
VI. WITHDRAWAL OF STANDARD			
Spec. for Lock-Bar Steel Pipe (A 137 – 34)	88	0	71
Spec. for Carbon-Steel Bars for Vehicle and General Purpose Springs (A 58 – 27)	83	1	68
VII. REAFFIRMATION OF STANDARDS			
Spec. for Open-Hearth Steel Girder Rails of Plain, Grooved, and Guard Types (A 2 – 27)	80	0	79
Spec. for Low-Carbon Steel Joint Bars (A 3 – 33)	80	0	79
Spec. for Medium-Carbon Steel Joint Bars (A 4 –14)	80	0	79
Spec. for Soft Steel Track Spikes (A 65 – 33)	80	0	79
Spec. for Steel Screw Spikes (A 66 – 33)	80	0	79
Spec. for Steel Tie Plates (A 67 – 33)	80	0	79
Spec. for Low-Carbon Steel Track Bolts and Nuts (A 76 – 33)	80	0	79
Spec. for Rail-Steel Bars for Concrete Reinforcement (A 16 – 35)	91	0	68
Spec. for Cold-Drawn Steel Wire for Concrete Reinforcement (A 82 – 34)	91	0	68
Spec. for Axle-Steel Bars for Concrete Reinforcement (A 160 – 39)	91	0	68
Spec. for Fabricated Steel Bar or Rod Mats for Concrete Reinforcement (A 184 – 37)	91	0	68
Spec. for Welded Steel Wire Fabric for Concrete Reinforcement (A 185 – 37)	91	0	68
Spec. for Carbon-Steel Axles for Cars and Tenders (A 21 – 36)	84	0	75
Spec. for Austenitic Manganese-Steel Castings (A 128 – 33)	91	0	68
Spec. for Forge-Welded Steel Pipe (A 136 – 34)	88	0	71
Spec. for Riveted Steel and Wrought-Iron Pipe (A 138 – 34)	88	0	71

REAFFIRMATION

The various subcommittees have reviewed their respective specifications in detail and unless noted later in the report, or unless changes are being recommended in a standard, the groups have determined that a standard which has stood for six years or more without revision are in line with present practice and recommended that the reaffirmation note appear on the items listed in Table I.

RECOMMENDATIONS AND DISCUSSION ON STANDARDS AND TENTATIVE STANDARDS

The detailed recommendations with respect to standards and tentative standards, listed in Table I, are set forth below grouped for ease of reference under appropriate headings which classify the materials covered. Comments as to the reasons for these recommendations are included wherever appropriate.

In the case of the revisions recommended for immediate adoption in several of the standards, the committee asks for a nine-tenths affirmative vote at the annual meeting in order that these recommendations may be referred to letter ballot of the Society. The standards, tentative standards, and tentative revisions appear in their present form in either the 1939 Book of Standards, Part I, or the 1940 and 1941 Supplements, Part I.

SPRING STEEL

Subcommittee IV

Standard Specifications for Chromium-Vanadium Steel Bars for Springs (**A 60 – 39**):

The following revision is recommended for immediate adoption to bring the chromium range in line with current practice and in closer agreement with existing standards:

Section 3.—Change the chromium range from "0.90 to 1.10" to read "0.80 to 1.10" per cent.

Standard Specifications for Carbon-Steel Bars for Vehicle and General Purpose Springs (**A 58 – 27**) *and for Carbon-Steel Bars for Springs* (**A 14 – 39**):

Consideration of these specifications by a special section resulted in a recommendation that Specifications A 58 be withdrawn, and that the analysis covered be included in Specifications A 14 – 39, as follows:

Section 1.—Revise to provide for addition of new Grade C, indicating that this is for applications where definitely specified.

Section 3.—Add the following chemical composition requirements for a new grade C:

	Grade C
Carbon, per cent	0.85 to 1.05
Manganese, per cent	0.25 to 0.50
Phosphorus, max., per cent	0.05
Sulfur, max., per cent	0.05

STEEL FORGINGS

Subcommittee VI

Subcommittee VI has reviewed each of its specifications and as indicated in Table I the adoption as standard of the four existing tentative specifications is recommended. Editorial clarification of the section on number of tests in each of the four specifications is being studied.

The Standard Specifications for Carbon-Steel Axles for Cars and Tenders (A 21 – 36) have been reaffirmed.

The Tentative Specifications for Carbon-Steel and Alloy-Steel Ring and Disk Forgings (A 243 – 41 T) and for Carbon-Steel and Alloy-Steel Blooms, Billets, and Slabs for Forgings (A 248 – 41 T), have been in existence only a year and are to continue in their present status. Specifications A 248 are essentially a revision of the existing Standard Specifications for Carbon-Steel and Alloy-Steel Blooms, Billets, and Slabs for Forgings (A 17 – 29). The situation in respect to the latter is complicated by the national emergency in connection with alloy steels. However, since the standard specifications as well as the tentative are still in use for export and domestic use, both are to be continued in the 1942 Book of A.S.T.M. Standards, Part I.

Several important Emergency Alternate Provisions as recommended by the National Emergency Steel Specifications Technical Advisory Committee on Axle and Railroad Forgings in the Tentative Specifications for Carbon-Steel Forgings for Locomotives and Cars (A 236 – 40 T) undoubtedly will be approved in the near future.[5] Among other matters, these provisions would set up two new classes of forgings with distinctly higher physical properties than now embodied.

STEEL WHEELS AND TIRES

Subcommittee VII

Proposed Emergency Alternate Provisions.—In addition to the Emergency Alternate Provision in the Standard Specifications for Steel Tires (A 26 – 39), Subcommittee VII on Rolled Steel Wheels and Steel Tires will request that emergency alternate provisions[5] be approved in the Standard Specifications for Wrought Steel Wheels for Electric Railway Service (A 25 – 41), for Multiple-Wear Wrought Steel Wheels (A 57 – 39), and for One-Wear and Two-Wear Wrought Steel Wheels (A 186 – 39), and in the Tentative Specifications for Heat-Treated Wrought Steel Wheels (A 244 – 41 T), providing that for acid steel a sulfur content of 0.06 per cent be permitted instead of the present requirement of 0.05 per cent.

Proposed Adoption as Standard of Tentative Standard.—It is recommended that the Tentative Specifications for Heat-Treated Wrought Steel Wheels (A 244 – 41 T) be approved for submission to letter ballot for advancement to standard. Although these specifications have been issued for only one year, no criticism has been received and, further, they were based on industrial practice which has been used for some time.

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[5] See Editorial Note, p. 94.

STEEL CASTINGS

Subcommittees VIII and XXII

Two subcommittees have in their charge specifications for steel castings, namely, Subcommittee VIII on Steel Castings which has five specifications and Subcommittee XXII on Valves, Fittings, Piping, and Flanges for High-Temperature and Subatmospheric-Temperature Service which has three specifications. As indicated previously in this report a number of emergency alternate provisions have been established in several of these standards. In three of the specifications, as indicated in Table I, tentative revisions are recommended for adoption, providing for the determination of yield strength since many grades of castings do not as a rule exhibit a definite yield point.

REVISION OF STANDARD, IMMEDIATE ADOPTION

Standard Specifications for Alloy-Steel Castings for Valves, Flanges, and Fittings for Service at Temperatures from 750 to 1100 F. (**A 157 – 41**):

In these specifications the silicon ranges of grades C5 A (4 to 6 per cent chromium) and C5 B (4 to 6 per cent chromium-silicon-molybdenum) are not contiguous. The silicon requirement of 0.50 max., per cent of grade C5 A and the range of 0.75 to 1.50 per cent of grade C5 B leaves a range of from 0.50 to 0.75 per cent silicon, in which a heat cannot be properly classified. Silicon up to 0.75 per cent helps the castability of grade C5 A without any deleterious effect on high-temperature physical properties. Above this, castability is no further improved, although the higher silicon content improves scaling resistance, at some expense to the high-temperature strength properties. The same situation exists with respect to grades C3 A and C3 B.

When Section 2 on Process was revised last year to include the added grade C12 (8 to 10 per cent Chromium-Molybdenum), it was included with the three other grades C 6, C 9, C 10 to be produced by the electric-furnace or crucible process. Further consideration has led to the conclusion that the open-hearth was excluded for these higher alloys, primarily because the practice at the time the specifications were drafted indicated use of the electric-furnace.

To incorporate the changes discussed above, to make other chemical requirements consistent, and to improve castability, the following revisions are recommended for immediate adoption as standard:

Table I.—Change the carbon content of grades C 3A, C 3B, C 5A, and C 5B from "0.35 max. per cent" in the first two grades and "0.15 to 0.35 per cent" in the last two grades to read "0.30 max. per cent" in all three grades.

Change the silicon content of grades C 3A and C 5A from "0.50" to "0.75" max., per cent.

Table II.—Change the minimum tensile strength of grades C 5A and C 5B from "100,000 psi." to read "90,000 psi." and the minimum yield point from "65,000 psi." to read "60,000 psi."

Section 2.—Change the last sentence of this section by the addition of the italicized words and the omission of those in brackets:

> Steels C6, C9, C10, and C12 shall be made by the electric-furnace or [crucible process] *other process approved by the purchaser.*

REVISIONS OF TENTATIVE STANDARDS

Tentative Specifications for Carbon-Steel Castings Suitable for Fusion Welding for Service at Temperatures up to 850 F. (**A 216 – 41 T**) *and for Alloy-Steel Castings Suitable for Fusion Welding for Service at Temperatures from 750 to 1100 F.* (**A 217 – 41 T**):

To bring the silicon requirements in Specifications A 216 in line with emergency provisions which were established, it is recommended that the present silicon requirement of "0.50" be changed to read "0.60" max., per cent. The same change is recommended in grades WC 1 and WC 2 in Specifications A 217.

PIPE AND TUBULAR PRODUCTS

Subcommittees IX and XXII

For several years there has been intensive activity involving the development of standard specifications for pipe and tubular products. This activity has continued during the past year and Subcommittee XXII on Valves, Fittings, Piping, and Flanges for High-Temperature and Subatmospheric-Temperature Service, which has under its jurisdiction three specifications covering pipe for use at elevated temperatures, and Subcommittee IX on Steel Tubing and Pipe, which has some 30 specifications in its charge for use at normal or reasonably elevated temperatures, have a number of important recommendations on standards as detailed below.

As indicated previously, the new Tentative Specifications for Welded and Seamless Steel Pipe Piles (A 252 – 42 T) were approved through the work of Subcommittee IX. This subcommittee has prepared proposed Tentative Specifications for Copper Brazed Steel Tubing[6] covering material suitable for general engineering uses, particularly in the automobile, refrigerator, and stove industries (fuel lines, brake lines, oil lines, heating and cooling units, etc.) in sizes from 3/16 to 7/16 in., inclusive, in outside diameter. New Specifications for Welded Alloyed Open-Hearth Iron Pipe, developed by a special section of Subcommittee IX, are also being submitted for publication as tentative.[6]

Affecting some of the specifications in both subcommittees have been intensive discussions of values for elongation and approval of a more rational basis for determining this important property; also, a new formula for determining flattening test requirements. Statements on these two matters appear as Appendices I and II. Another matter which is still being discussed is the inclusion of the acid-bessemer process for producing seamless pipe.

As detailed below, changes in the widely used Standard Specifications for Welded and Seamless Steel Pipe (A 53 – 40) are recommended, involving elongation values, flattening test, use of acid-bessemer steel, and fabrication by the electric-resistance process. Some of these changes are similarly being made in the specifications listed in the following paragraph.

The Standard Specifications for Lap-Welded and Seamless Steel Pipe (A 106 – 41), and the Tentative Specifications for Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F. (A 158 – 41 T), and for Seamless Carbon-Molybdenum Alloy-Steel Pipe for Service at Temperatures from 750 to 1000 F. (A 206 – 41 T), under the jurisdiction of Subcommittee XXII, are very widely used. Because of the very severe operating conditions and changing concepts of how best to handle various requirements, revisions have been proposed yearly in the specifications and a number of recommendations are being made this year to improve portions of the standards.

ADOPTION AS STANDARD OF EXISTING TENTATIVE REVISIONS

All of the revisions in the ten specifications covering pipe and tubular products published for a year or more are recommended for adoption as standard, as indicated in Table I.

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[6] These specifications were accepted as tentative by the Society and appear in the 1942 Book of A.S.T.M. Standards, Part I, pp. 1044 and 1056.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption as standard the revisions given below in the various standards listed:

Standard Specifications for Seamless Cold-Drawn Low-Carbon Steel Heat-Exchanger and Condenser Tubes (**A 179 – 40**):

In order to bring Section 5 on Check Analysis in line with the basic Specifications for Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes (A 83 – 40) providing that check analysis may be made by the purchaser rather than the manufacturer, the following change is recommended:

Section 5.—Change from its present form to read as follows by the addition of the italicized words and the omission of those in brackets:

> 5. (*a*) [When requested by the purchaser,] An analysis of two tubes from each lot of 250 tubes or fraction thereof [shall] *may* be made *by the purchaser*. Drillings for analysis shall be taken from several points around each tube selected for analysis. The chemical composition thus determined shall conform to the requirements specified in Section 4.

Standard Specifications for Electric-Resistance-Welded Steel Heat-Exchanger and Condenser Tubes (**A 214 – 40**):

For the same reason given above for Specifications A 179 – 40, the following change is recommended:

Section 5.—Change from its present form to read as follows by the addition of the italicized words and the omission of those in brackets:

> 5. (*a*) [When requested by the purchaser,] An analysis of two tubes from each lot of 250 tubes or fraction thereof [shall] *may* be made *by the purchaser*. Drillings for analysis shall be taken from [at least four] *several* points around each tube selected for analysis. The chemical composition [of the composite] thus determined shall conform to the requirements specified in Section 4.

Standard Specifications for Welded and Seamless Steel Pipe (**A 53 – 40**):

In Appendix I there appears a condensed discussion on studies made by a section concerning the effect of wall thickness on elongation of pipe and the development of specification values based on wall thickness. Also in Appendix II is a statement on a proposed formula and new procedure for determining flattening test requirements, which has been studied in a special section of Subcommittees IX and XXII. Since a considerable tonnage of steel for seamless pipe has been made for a number of years by the acid-bessemer process, with apparently satisfactory service resulting, the use of this process in Standard A 53 is being recommended. To effect all of these proposals and further to provide for the inclusion of the electric-resistance-welded process and to indicate that the material is suitable for fusion welding, the following changes are recommended for immediate adoption:

Section 1 (*a*).—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

> (*a*) These specifications cover black and hot-dipped-galvanized [furnace] welded and seamless steel pipe. Pipe ordered under these specifications is intended for coiling, bending, flanging, and other special purposes, *and is suitable for fusion welding*. Butt-welded pipe is not intended for flanging. The purposes for which the pipe is intended should be stated in the order.[a] When seamless *or electric-resistance-welded* pipe is ordered for close coiling, cold bending, or for forge welding, grade A [seamless] should be specified rather than grade B [seamless].

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[a] *Furnace Double Welding.*—Unless furnace-double-welded pipe is specifically called for in the order, single-welded pipe may be furnished. When furnace-double-welded pipe is ordered, the flattening test on crop ends specified in Section 10 shall be made with the weld on the side.

Section 2 (*a*).—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

2. (*a*) The steel for [welded pipe] *both welded and seamless pipe* shall be [soft weldable quality] made by one or more of the following processes: open-hearth, electric-furnace, or acid-bessemer. The steel for [seamless pipe shall be made by either of the following processes: open-hearth or electric-furnace] *furnace-welded pipe shall be of soft weldable quality.*

TABLE II.—TENSILE REQUIREMENTS.

	Furnace Welded		Seamless or Electric-Resistance-Welded	
	Acid-Bessemer	Open-Hearth or Electric-Furnace	Grade A	Grade B
Tensile strength, min., psi	50 000	45 000	48 000	60 000
Yield point, min., psi	30 000	25 000	30 000	35 000
Elongation in 8 in., min., per cent	18[a]	20[a]	....	....
Elongation in 2 in., min., per cent	....	30[b]	....	....
Elongation in 2 in., min., per cent:				
Basic minimum elongation for walls 5⁄16 in. and over in thickness, longitudinal strip tests, and for all small sizes tested in full section	....	....	35	30
When standard round 2-in. gage length test specimen is used	....	....	28	22
For longitudinal strip tests a deduction for each 1⁄32 in. decrease in wall thickness below from the 5⁄16 in. from the basic minimum elongation of the following percentage	....	....	1.75[c]	1.50[c]

[a] Gage distances for measuring elongation on welded pipe of nominal sizes ¾ in. and smaller shall be as follows:

Nominal Size, in.	Gage Length, in.
¾ and ½	6
⅜ and ¼	4
⅛	2

[b] When specimen shown in Fig. 1 is used; see Section 9 (*a*).

[c] The following table gives the computed minimum values:

Wall Thickness, in.	Elongation, min., per cent	
	Grade A	Grade B
5⁄16 (0.3125)	35.00	30.00
9⁄32 (0.281)	33.25	28.50
¼ (0.250)	31.50	27.00
7⁄32 (0.2187)	29.75	25.50
3⁄16 (0.1875)	28.00	24.00
5⁄32 (0.156)	26.25	22.50
⅛ (0.125)	24.50	21.00
3⁄32 (0.0937)	22.75	19.50
1⁄16 (0.0625)	21.00	18.00

(*b*) *Furnace*-welded pipe 3 in, and under in nominal diameter may be butt-welded, unless otherwise specified. *Furnace*-welded pipe over 3 in. in nominal diameter shall be lap-welded.

Section 3.—Change to read as follows by the addition of the italicized words and figures and the omission of those in brackets.

3. [Open-hearth or electric-furnace.] *The* steel shall conform to the following ladle analysis requirement as to chemical composition:

	Lap-Welded	Seamless *or Electric-Resistance-Welded*
Phosphorus, max., per cent:		
Open-hearth or electric-furnace	0.06	0.045
Acid-bessemer	....	*0.11*

Section 4.—Change from its present form to read as follows by the addition of the italicized words:

4. An analysis of two pipes from each lot of 500 lengths or fraction thereof may be made by the purchaser. Drillings for analysis shall be taken from several points around each pipe selected for analysis. The phorphorus content *of open-hearth or electric-furnace steel* thus determined shall not exceed that specified in Section 3 by more than 25 per cent. *For acid-bessemer seamless or electric-resistance-welded*

steel pipe, the phosphorus content shall not exceed the maximum specified in Section 3.

Table I.—Modify the elongation requirements on seamless grades A and B by changing the table from its present form to read as shown in the accompanying Table II.

Table II.—In footnote *a* of this table a formula determines the hydrostatic test pressure for larger pipe. Agreement has been reached by the manufacturers so the committee recommends that the value of "*S*," fiber stress, in the formula $P = \frac{2St}{D}$ be changed from its present fixed value of "12,000 psi." to read "60 per cent of the specified yield point." This increases the test pressure considerably.

Section 7.—Add a new Paragraph (*c*) to read as follows, relettering the present Paragraph (*c*) as (*d*):

> (*c*) For electric-resistance-welded pipe, the test section shall be 4 to 6 in. in length and the weld shall be located 90 deg. from the line of direction of the applied force.

Reletter present Paragraph (*d*) to read (*e*), and change to read as follows:

> (*e*) The test shall consist in flattening a section of pipe between parallel plates until the opposite walls meet. For welded pipe, no opening in the weld shall take place until the distance between the plates is less than three-fourths of the original outside diameter for butt-weld, or two-thirds the outside diameter for lap-weld and electric-resistance-weld, and no cracks or breaks in the metal elsewhere than in the weld shall occur until the distance between the plates is less than shown below. For seamless pipe, no breaks or cracks in the metal shall occur until the distance between the plates is less than that shown below:

Kind of Pipe	Distance Between Plates "*S*"
For butt-welded pipe	60 per cent of outside diameter
For lap-welded pipe	one-third the outside diameter
For electric-resistance-welded pipe, grades A and B	one-third the outside diameter
For seamless pipe, grades A and B	to the distance "*S*" developed by the following formula:

$$S = \frac{(1 + e)t}{e + t/D}$$

where:

S = distance between flattening plates in inches,

t = nominal wall thickness of pipe in inches,

D = actual outside diameter of pipe in inches, and

e = deformation per unit length (constant for a given grade of steel, 0.09 for grade A and 0.07 for grade B).

Section 10 (b).—Delete the last phrase reading: "for lap-welded pipe."

Section 17.—Change from its present form to read as follows by the addition of the italicized words:

> 17. Each length of pipe shall be legibly marked by rolling, stamping, or stenciling to show the name or brand of the manufacturer; the type of pipe (that is, lap-welded, *electric-resistance-welded A*, *electric-resistance-welded B*, seamless A, or seamless B; *where acid-bessemer steel is used in seamless or electric-resistance-welded pipe, the word "bessemer" shall be added before the letter A or B*); XS for extra strong, XXS for double extra strong; ASTM A 53; the length; and the hydrostatic test pressure in pounds per square inch; except that for small diameter pipe which is bundled, this information may be marked on a tag securely attached to each bundle.

Standard Specifications for Black and Hot-Dipped Zinc-Coated (Galvanized) Welded and Seamless Steel Pipe for Ordinary Uses (**A 120 – 40**):

Section 2 (*a*).—To provide for the inclusion of acid-bessemer seamless steel pipe, change from its present form: namely,

> (*a*) The steel for welded pipe shall be of soft weldable quality made by one or more of the following processes: open-hearth, electric-furnace, or acid-bessemer. The steel for seamless pipe shall be made by either or both of the following processes: open-hearth, or electric-furnace.

to read as follows:

> (*a*) The steel for both welded and seamless pipe shall be made by one or more of the following processes: open-hearth, electric-furnace, or acid-bessemer. The steel for welded pipe shall be of soft weldable quality.

Table I.—Make the same change as recommended above in Table II of Specifications A 53 – 40.

Standard Specifications for Seamless Alloy-Steel Boiler and Superheater Tubes (**A 213 – 40**):

The situation with respect to chemical composition of corrosion-resisting chromium nickel sheet strip and plate is changing because of the scrap situation and related factors resulting from the emergency. However, the committee feels the changes in Table II, given below, will bring them more nearly in line with current practice. It is quite possible that additional changes will be forthcoming in the near future.

Table II.—To bring the austenitic steel grades T 18 (chromium-nickel-titanium) and T 19 (chromium-nickel-columbium) in line with the Standard Specifications for Corrosion-Resisting Chromium-Nickel Steel Sheet, Strip, and Plate for Fusion-Welded Unfired Pressure Vessels (A 240 – 41), change the chemical requirements of grades T 18 and T 19 to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

	Grade T 18	Grade T 19
Carbon, max., per cent	0.08	0.08
Manganese, max., per cent	[3.00] *2.50*	[3.00] *2.50*
Phosphorus, max., per cent	[0.03] *0.035*	[0.03] *0.035*
Sulfur, max., per cent	0.03	0.03
Silicon, max., per cent	0.75	0.75
Nickel, ***min.***, per cent	[9.00 to 13.00] *9.00*	[9.00 to 13.00] *9.50*
Chromium, ***min.***, per cent	[17.00 to 20.00] *17.00*	[17.00 to 20.00] *17.00*
Titanium, per cent	[0.35][a]	....
Columbium, per cent	....	[0.70][b]

[a] ***Grade T 18 shall have a titanium content of not less than five times the carbon content and not more than 0.60 per cent.***

[b] ***Grade T 19 shall have a columbium content of not less than ten times the carbon content and not more than 1.00 per cent.***

ADOPTION OF TENTATIVE STANDARDS AS STANDARDS

The committee recommends for adoption as standard the two following tentative specifications, the first named with revisions as indicated below.

Tentative Specifications for Atomic-Hydrogen-Arc-Welded and Electric-Resistance-Welded Alloy-Steel Boiler and Superheater Tubes (**A 249 – 41 T**) *and Tentative Specifications for Seamless Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes* (**A 209 – 41 T**).

Specifications A 209, covering three grades of seamless-carbon-molybdenum tubes, were issued in 1938 and revised in the two subsequent years. Since no major criticisms have been received, adoption as standard is deemed in order.

For the reason as discussed above under Specifications A 213 – 40, changes in Specifications A 249 are proposed for adoption as standard involving Table I by which the same changes will be made in Table I, grades T 18 and T 19, as recommended above in Table II of Specifications A 213.

REVISIONS OF TENTATIVE STANDARDS

The committee recommends revisions in the following tentative standards:

Tentative Specifications for Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F. (**A 158 – 41 T**):

Section 8 (a).—Revise the elongation requirements by changing the table from its present form to read as shown in the accompanying Table **III**.

Section 10.—Make the same changes in the flattening test requirements as are recommended in Section 7 of Specifications A 106 – 41, except that this specification does not cover grades A and B pipe and the value of "*e*" shall be 0.08 for ferritic steels and 0.09 for austenitic steels.

Tentative Specifications for Seamless Carbon-Molybdenum Alloy-Steel Pipe for Service at Temperatures from 750 to 1000 F. (**A 206 – 41 T**):

Section 2.—Delete Paragraphs (*d*) and (*e*) and add a new Paragraph (*d*) to read as follows:

> (*d*) When requested by the purchaser and so stated in the order, hot-finished pipe in the as-finished condition in nominal sizes 3 in. and over made to schedules 140 and 160 shall have a structural grain size[a] which is predominately 3 to 6 as measured on an A.S.T.M. grain size chart.

Section 8 (a).—Revise the elongation requirements by changing the table from its present form to read as shown in the accompanying Table IV.

---

[a] The term "structural grain size" refers to the predominant grain as evidenced by ferrite grains, Widmänstatten areas, or pearlite patches.

Section 10.—Make the same changes in the flattening test requirements as are recommended in Section 7 of Specifications A 106 – 41, except that this specification does not cover grades A and B pipe and the value of "*e*" shall be 0.08.

Section 12.—Change from its present form to read as follows:

> 12. When requested by the purchaser and so stated in the order, the manufacturer shall furnish one photomicrograph at 100 diameters from a specimen of pipe in the as-finished condition for each individual size and wall thickness from each melt, for pipe in nominal sizes 3 in. and over made to schedules 140 and 160, to demonstrate that such pipe conforms to the requirements of Section 2 (*d*) when the order states that those requirements are to be met. Such photomicrographs shall be suitably identified as to pipe size, wall thickness, and melt. No photomicrographs for the individual pieces purchased shall be required except as specified in Paragraph S-7.

Supplementary Requirements, Section S7.—Change from its present form to read as follows:

> S7. In addition to the photomicrographs required in accordance with Section 12, for hot-finished pipe in nominal sizes 3 in. and over made to schedules 140 and 160, and specified to conform to the requirements of Section 2 (*d*), the purchaser may specify that photomicrographs shall be furnished from each end of one or more pieces from each lot of pipe in the as-finished condition. The purchaser shall state in the order the number of pipes to be tested from each lot. When photomicrographs are required on each piece, the photomicrographs from each lot of pipe in the as-finished condition which may be required under Section 12 may be omitted. All photomicrographs required shall be properly identified as to heat number, size, and wall thickness of pipe from which the section was taken. Photomicrographs shall be further identified to permit association of each photomicrograph with the individual piece of pipe it represents.

TABLE III.

	Ferritic		Austenitic	
Tensile strength, min., psi	60 000		75 000	
Yield point, min., psi	30 000		30 000	
	Longitudinal	Transverse	Longitudinal	Transverse
Elongation in 2 in., min., per cent:				
Basic minimum elongation for walls 5/16 in. and over in thickness, strip tests, and for all small sizes tested in full section	30	20	50	40
When standard round 2-in. gage length test specimen is used	22	14	35	25
For strip tests, a deduction for each 1/32 in. decrease in wall thickness below 5/16 in. from the basic minimum elongation of the following percentage	1.50[a]	1.00[a]	2.50[a]	2.00[a]

[a] The following table gives the calculated minimum values:

Wall Thickness, in.	Ferritic		Austenitic	
	Longitudinal	Transverse	Longitudinal	Transverse
5/16 (0.3125)	30.00	20.00	50.00	40.00
9/32 (0.281)	28.50	19.00	47.50	38.00
1/4 (0.250)	27.00	18.00	45.00	36.00
7/32 (0.2187)	25.50	....	42.50	....
3/16 (0.1875)	24.00	....	40.00	....
5/32 (0.156)	22.50	....	37.50	....
1/8 (0.125)	21.00	....	35.00	....
3/32 (0.0937)	19.50	....	32.50	....
1/16 (0.0625)	18.00	....	30.00	....

REVERSION OF STANDARD TO TENTATIVE

Standard Specifications for Lap-Welded and Seamless Steel Pipe for High-Temperature Service (**A 106 – 41**):

To incorporate new elongation values for seamless pipe, and to provide for the use of acid bessemer steel and the new flattening test requirements, the following revisions are proposed following reversion of the specifications to the status of tentative. The latter action is felt justifiable because of the great importance of some of the changes, and it was deemed advisable to incorporate them in the specifications immediately.

TABLE IV

Tensile strength, min., psi	55 000	
Yield point, min., psi	30 000	
	Longitudinal	Transverse
Elongation in 2 in., min., per cent:		
Basic minimum elongation for walls 5/16 in. and over in thickness, strip tests, and for all small sizes tested in full section	30	20
When standard round 2-in. gage length test specimen is used	22	14
For strip tests, a deduction for each 1/32 in. decrease in wall thickness below 5/16 in. from the basic minimum elongation of the following percentage	1.50[a]	1.00[a]

[a] The following table gives the calculated minimum values:

Wall Thickness, in.	Longitudinal	Transverse
5/16 (0.3125)	30.00	20.00
9/32 (0.281)	28.50	19.00
1/4 (0.250)	27.00	18.00
7/32 (0.2187)	25.50	....
3/16 (0.1875)	24.00	....
5/32 (0.156)	22.50	....
1/8 (0.125)	21.00	....
3/32 (0.0937)	19.50	....
1/16 (0.0625)	18.00	....

Section 2 (a).—Change the last sentence to read as follows by the addition of the italicized words and the omission of those in brackets:

> All seamless pipe shall be made of killed steel by [either or both] *one or more* of the following processes: open-hearth, [or] electric-furnace, *or acid-bessemer.*

Table I.—Place a footnote a after the values of phosphorus, max. per cent for both grades A and B seamless and add the following as footnote a:

> When acid-bessemer steel is furnished for grades A or B seamless pipe, the phosphorus content shall be 0.11, max., per cent and the carbon shall be 0.25 per cent.

Section 7.—Change Paragraph (*b*) to read as follows:

> (*b*) For grades A and B seamless pipe over 2 in. in nominal diameter a section of pipe not less than 2½ in. in length shall be flattened cold between parallel plates until the opposite walls of the pipe meet. No cracks or breaks in the metal shall occur until the distance between the plates is less than that calculated for the value of "*S*" by the following formula:
>
> $$S = \frac{(1 + e)\,t}{e + t/D}$$
>
> where:
>
> S = distance between flattening plates in inches.
>
> t = nominal wall thickness of pipe in inches,
>
> D = actual outside diameter of pipe in inches, and
>
> e = deformation per unit length (constant for a given grade of steel, 0.09 for grade A and 0.07 for grade B).
>
> Evidence of laminations or burnt material shall not develop during the entire flattening process.

Omit Paragraph (*c*) since reference to grade B is included in new Paragraph (*b*), above.

Section 17.—Change the first sentence to read as follows by the addition of the italicized words and the omission of those in brackets:

> Each length of pipe manufactured in accordance with these specifications shall be legibly marked *beginning within 12 in. from the end,* either by stenciling, stamping, or rolling, with the manufacturer's private identifying mark, together with the designation A 106-A (or B depending on the grade of seamless steel used, or no letter if lap-welded; *where acid-bessemer steel is used, the word bessemer shall be added, that is, seamless bessemer A or seamless bessemer B*), the hydrostatic test pressure, and an additional S if the pipe conforms *in any case* to the supplementary requirements specified in Paragraphs S1 to S6.

Table II.—Modify the elongation requirements on seamless grades A and B by changing the table from its present forms to read as shown in the accompanying Table V.

EDITORIAL CHANGES

To clarify the use of the standard round 2-in. gage length tension test specimen, an editorial change is being made in Sections 9 (*b*) and (*c*) of Standard Specifications for Lap-Welded and Seamless Steel Pipe (A 106 – 41), 12 (*b*) and (*c*) of Tentative Specifications for Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F. (A 158 – 41 T), and 13 (*b*) and (*c*) of Tentative Specifications for Seamless Carbon-Molybdenum Alloy-Steel Pipe for Service at Temperatures from 750 to 1000 F. (A 206 – 41 T) to provide for inclusion of the ¾-in. pipe wall thickness by changing the words "exceeds ¾ in." to read "¾ in. and over" where it appears in each of these paragraphs; also it is being clearly indicated that this specimen may be used.

Since certain pipe size schedule numbers are not normally produced but for which dimensional requirements are given in tables in each of the above three specifications, editorial changes are being made to delete the nominal wall thickness values in Schedules 10, 20, and 30 for pipe 30 in. in outside diameter; in Schedules 140 and 160 for pipe 24 in. in outside diameter; and in Schedule 160 for pipe 20 in. in outside diameter. These changes will be accompanied by editorial modifications of certain notes accompanying the tables.

TABLE V.—TENSILE REQUIREMENTS.

	Welded Open-Hearth	Seamless			
		Grade A (Note 2)		Grade B Silicon-Killed	
Tensile strength, min., psi	45 000	48 000		60 000	
Yield point, min., psi	25 000	30 000		35 000	
Elongation in 8 in., min., per cent	22	....		....	
		Longitudinal	Transverse	Longitudinal	Transverse
Elongation in 2 in., min., per cent:					
Basic minimum elongation for walls 5/16 in. and over in thickness, strip tests, and for all small sizes tested in full section	....	35	25	30	16.5
When standard round 2-in. gage length test specimen is used	....	28	20	22	12
For strip tests, a deduction for each 1/32 in. decrease in wall thickness below 5/16 in. from the basic minimum elongation of the following percentage	....	1.75[a]	1.25[a]	1.50[a]	1.00[a]

[a] The following table gives the computed minimum values:

Wall Thickness, in.	Elongation, min. per cent			
	Grade A		Grade B	
	Longitudinal	Transverse	Longitudinal	Transverse
5/16 (0.3125)	35.00	25.00	30.00	16.50
9/32 (0.281)	33.25	23.75	28.50	15.50
1/4 (0.250)	31.50	22.50	27.00	14.50
7/32 (0.2187)	29.75	....	25.50	....
3/16 (0.1875)	28.00	....	24.00	....
5/32 (0.156)	26.25	....	22.50	....
1/8 (0.125)	24.50	....	21.00	....
3/32 (0.0937)	22.75	....	19.50	....
1/16 (0.0625)	21.00	....	18.00	....

The section on Marking in the above three specifications is being revised editorially by changing the sentence reading

"Marking shall be within 12 in. of one end of each length" to read "Marking shall begin within 12 in. of one end of each length."

BOILER STEELS

Subcommittee XI

The revisions in the following standards are recommended for immediate adoption as standard:

Standard Specifications for Molybdenum-Steel Plates for Boilers and Other Pressure Vessels (**A 204 - 39**):

Table I.—Change the manganese requirement in grades A, B, and C from the present range of "0.50 to 0.90" to read "0.90, max.", per cent.

This change is being made because it is advisable to keep the manganese under 0.50 per cent on light gage plates in order to stay within the tensile ranges enumerated, particularly on grade A.

Standard Specifications for Carbon-Steel Plates for Stationary Boilers and Other Pressure Vessels (**A 70 - 39**):

Section 1.—Revise to limit the thickness of flange quality plates covered by the specifications to be 2 in. max., and for firebox quality to be 4 in.

This addition brings Specifications A 70 in line with the two other widely used plate specifications A 201 - 39 and A 212 - 39.

Standard Specifications for Low-Carbon Nickel-Steel Plates for Boilers and Other Pressure Vessels (**A 203 - 39**):

Section 1.—Add a new sentence at the end of this section to read as follows:

> This material is suitable for fusion welding.

It is understood that the A.S.M.E. Boiler Code Specification S-43 which is identical with the A.S.T.M. Specifications A 203 - 39 will be changed, thus providing for the use of this material in welding fabrication.

COMMERCIAL BAR STEELS

Subcommittee XV

Standard Specifications for Commercial Quality Hot-Rolled Bar Steels (**A 107 - 40**):

The committee recommends for immediate adoption as standard the following changes in Table III affecting the last bracket for rounds $5\frac{1}{2}$ to 8 in. and in Table V to provide an additional column for flats under $\frac{1}{4}$ in. in thickness, in order that certain permissible variations may be in line with latest practice. The need for the change in Table V arises from a commercial reclassification of flat bars which now run down to 0.203 in. in thickness instead of 0.250 in.

Table III.—Omit the last line and add the following material:

Specified Size, in.	Variations from Size, in.		Out-of-Round or Square, in.
	Over	Under	
Over 5½ to 6½, incl.	⅛	0	0.070
Over 6½ to 8, incl.	5/32	0	0.085

Table V.—Add a new column to cover material under $\frac{1}{4}$ in. in thickness reading as follows:

Under ¼
0.006
0.008
0.010
0.010

Change the Note under this table to show that minimum thickness of flat bars is $\frac{13}{64}$ in. instead of $\frac{1}{4}$ in.

Subcommittee XV on Commercial Bar Steels, which is responsible for the Specifications for Commercial Quality Hot-Rolled Bar Steels (A 107 - 40) and also for the Specifications for Commercial Cold-Finished Bar Steels and Cold-Finished Shafting (A 108 - 36), has discussed at two meetings the status of the chemistry requirements, and in March considered drafts of revised tables to bring the grades covered in line with latest decisions reached jointly by the

Society of Automotive Engineers and the American Iron and Steel Institute. Action on a proposal to recast the grades of hot-rolled and cold-finished bars in these two specifications was deferred in view of probable further simplification by government agencies. Meanwhile comparison of the present compositions with the AISI-SAE grades indicates the committee's selection of significant chemical grades was a pretty good job of pioneering and practically all will have their counterparts in the current standardization.

SHEET STEEL

Subcommittee XIX

Tentative Specifications for Light Gage Structural Quality Flat Hot-Rolled Carbon Steel (0.2499 and 0.1874 in. to 0.0478 in. in Thickness) (**A 245 – 41 T**):

To take care of a discrepancy in the test specimen section of this specification, issued as tentative last year, and to make it complete, it is recommended that the following changes be made:

Section 7 (a).—Insert in the table for the three grades A, B, and C, the following minimum values for elongation in 2 in. of 29, 25, and 22 per cent, respectively.

Section 9 (b).—Change from its present form to read as follows:

> (*b*) Tension test specimens shall have their longest axis in the direction of the last hot-rolling and shall be the full thickness of the material as rolled. Test specimens may be machined to the form and dimensions shown in Fig. 1 or 2 as specified.

Figs. 1 and 2.—Add as Fig. 1 the standard rectangular test specimen with 8 in. gage length and as Fig. 2 the standard rectangular test specimen with 2 in. gage length. These are the same as Fig. 1 and 2 which appear in the Tentative Methods of Tension Testing of Metallic Materials (E 8 – 40 T).[7]

Tentative Specifications for Light Gage Structural Quality Flat Rolled Carbon Steel (0.0477 to 0.0225 in. in Thickness) (**A 246 – 41 T**):

To make these specifications all inclusive, it is recommended that a figure (Fig. 2, showing the standard rectangular (flat) 2-in. gage length specimen, appearing in the Tentative Methods of Tension Testing of Metallic Materials (E 8 – 40 T)[7]) be incorporated showing the test specimen dimensions, which will also involve modifications of Section 9 (*b*).

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, N. L. Mochel.
Vice-Chairman, H. B. Oatley.
Vice-Chairman, E. F. Kenny.
Secretary, H. P. Bigler.

This report has been submitted to letter ballot of the committee which consists of 230 members; 134 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

N. L. MOCHEL,
Chairman.

H. P. BIGLER,
Secretary.
R. J. PAINTER,
Staff Assistant.

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[7] 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 453.

EDITORIAL NOTE

Subsequent to the annual meeting, the following Emergency Alternate Provisions, submitted by Committee A-1, were accepted by Committee E-10 on Standards on the date indicated:

DESIGNATION	EMERGENCY ALTERNATE PROVISIONS IN	ISSUED
	Standard Specifications for:	
EA - A 21	Carbon-Steel Axles for Cars and Tenders (A 21 - 36)	Oct. 5, 1942
EA - A 236	Carbon-Steel Forgings for Locomotives and Cars (A 236 - 42)	Aug. 12, 1942
EA - A 238	Alloy-Steel Forgings for Locomotives and Cars (A 238 - 42)	Oct. 5, 1942
EA - A 25	Wrought Steel Wheels for Electric Railway Service (A 25 - 41)	June 22, 1942
EA - A 25a	Wrought Steel Wheels for Electric Railway Service (A 25 - 41)	Aug. 24, 1942
EA - A 57	Multiple-Wear Wrought Steel Wheels (A 57 - 39)	June 22, 1942
EA - A 186	One-Wear and Two-Wear Wrought Steel Wheels (A 186 - 39)	June 22, 1942
EA - A 244	Heat-Treated Wrought Steel Wheels (A 244 - 42)	June 22, 1942
EA - A 120	Black and Hot-Dipped Zinc-Coated (Galvanized) Welded and Seamless Steel Pipe for Ordinary Uses (A 120 - 42)	Aug. 24, 1942
EA - A 134	Electric-Fusion-Welded Steel Pipe (Sizes 30 in. and Over) (A 134 - 42)	Aug. 18, 1942
EA - A 139	Electric-Fusion-Welded Steel Pipe (Sizes 8 in. to but not Including 30 in.) (A 139 - 42)	Aug. 18, 1942
EA - A 135	Electric-Resistance-Welded Steel Pipe (A 135 - 42)	Aug. 24, 1942
EA - A 211	Spiral-Welded Steel or Iron Pipe (A 211 - 40)	Aug. 18, 1942
EA - A 83	Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes (A 83 - 42)	Aug. 18, 1942
EA - A 161	Seamless Low-Carbon and Carbon-Molybdenum Steel Still Tubes for Refinery Service (A 161 - 40)	Aug. 18, 1942
EA - A 178	Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 - 40)	Aug. 18, 1942
EA - A 179	Seamless Cold-Drawn Low-Carbon Steel Heat-Exchanger and Condenser Tubes (A 179 - 42)	Aug. 18, 1942
EA - A 187	Seamless Cold-Drawn Alloy-Steel (4 to 6 per cent Chromium) Heat-Exchanger and Condenser Tubes (A 187 - 40)	Aug. 18, 1942
EA - A 188	Seamless Alloy-Steel (4 to 6 per cent Chromium) Still Tubes for Refinery Service (A 188 - 40)	Aug. 18, 1942
EA - A 192	Seamless Steel Boiler Tubes for High-Pressure Service (A 192 - 40)	Aug. 18, 1942
EA - A 199	Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes (A 199 - 40)	Aug. 18, 1942
EA - A 200	Seamless Alloy-Steel, Intermediate, Still Tubes for Refinery Service (A 200 - 40)	Aug. 18, 1942
EA - A 209	Seamless Carbon-Molybdenum Alloy-Steel. Boiler and Superheater Tubes (A 209 - 42)	Aug. 18, 1942
EA - A 213	Seamless Alloy-Steel Boiler and Superheater Tubes (A 213 - 42)	Aug. 24, 1942
EA - A 214	Electric-Resistance-Welded Steel Heat-Exchanger and Condenser Tubes (A 214 - 42)	Aug. 18, 1942
EA - A 226	Electric-Resistance-Welded Steel Boiler and Superheater Tubes for High-Pressure Service (A 226 - 40)	Aug. 18, 1942
EA - A 249	Atomic-Hydrogen-Arc-Welded and Electric-Resistance-Welded Alloy-Steel Boiler and Superheater Tubes (A 249 - 42)	Aug. 24, 1942
	Tentative Specifications for:	
EA - A 250	Electric-Resistance-Welded Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 250 - 41 T)	Aug. 24, 1942

These emergency provisions have been published in the ASTM BULLETIN and have also been issued in the form of pink stickers for attachment to the standards to which they apply.

Committee A-1 also submitted to Committee E-10 a revision of the Emergency Specifications for Carbon-Chromium Ball and Roller-Bearing Steels (ES-5). This recommendation was accepted by Committee E-10 on September 16, 1942, and the revised emergency specifications issued under the designation ES-5a appear in the 1942 Book of A.S.T.M. Standards, Part I, p. 954.

On August 24, 1942, Committee E-10 accepted the recommendation of Committee A-1 that the following standards be wthdrawn together with EA - A 187 and EA - A 188:

Standard Specifications for:

Seamless Cold-Drawn Alloy-Steel (4 to 6 per cent Chromium) Heat-Exchanger and Condenser Tubes (A 187 - 40), and

Seamless Alloy-Steel (4 to 6 per cent Chromium) Still Tubes for Refinery Service (A 188 - 40).

APPENDIX I

STATEMENT ON ELONGATION REQUIREMENTS FOR STEEL PIPE

Based on recommendations from Subcommittees IX on Steel Tubing and Pipe and XXII on Valves, Fittings, Piping and Flanges for High-Temperature and Subatmospheric-Temperature Service, Committee A-1 on Steel is asking the annual meeting of the Society to approve for reference to letter ballot for immediate adoption proposed revisions in two of the most important A.S.T.M. specifications for pipe: namely, the Standard Specifications for Welded and Seamless Steel Pipe (A 53 – 40) and for Lap-Welded and Seamless Steel Pipe for High-Temperature Service (A 106 – 41). These revisions, when adopted, will provide that the required elongation values be related to the thickness of the material. A joint committee from the two subcommittees has studied this matter and approved a statistical analysis submitted by H. R. Redington of the National Tube Co., chairman of the joint group. Since this recommendation is very significant, this statement is appended to the report so that full information will be available to those who are concerned with the matter. The recommendations affecting the two standards are published previously in this report.[1]

Present Requirements and Discussion:

In the two specifications under consideration, the tension test specimens are usually cut longitudinally from the pipe and not flattened between gage lengths, although if desired the test specimens may consist of a full section of the pipe up to the capacity of the testing machine. When impracticable to use the full wall thickness, the A.S.T.M. standard 2-in. gage length tension test specimen, commonly termed the 0.505-in. round test specimen, may be used. The strip specimens have a width of 1½ in. and a gage length usually of 8 in.; for seamless pipe it is 2 in. It is obvious that with the strip specimen the thickness may vary from the very thinnest walls manufactured to walls well over 1 in., thus presenting a wide variation in cross-sectional area with the gage length remaining the same.

It is well known that for a given grade of steel the percentage of elongation obtained depends on the relation between the cross-sectional area and the gage length. With a constant gage length, a reduction in cross-section causes a reduction in percentage of elongation. Because of this, it is necessary to provide elongation requirements varying with the wall thickness, if the same grade of material is to be obtained in all cases.

In connection with this problem of specifying elongation in relation to wall thickness (or cross-section area of specimen), it is significant that manufacturers and consumers recognize this principle in other A.S.T.M. specifications; for example, the Standard Specifications for Low Tensile Strength Carbon-Steel Plates of Structural Quality for Welding (A 78 – 40) in which a strip test specimen is used that is not at all unlike a strip test section from a pipe or tube.

Statistical Analysis:

To show the effect of variations in wall thickness on the elongation values a statistical analysis was made of over 7500 tension tests, the data being submitted by six pipe manufacturers as follows: Youngstown Sheet and Tube Co., Republic Steel Corp., Jones & Laughlin Steel Corp., Spang Chalfant Division of National Supply Co., National Tube Co., and Pittsburgh Steel Co. Tabulations of the resulting data, as applying to grades A and B pipe for which the tensile properties are the same in both Specifications A 53 and A 106, are given in the accompanying Tables I and II. (These data are for the most part from tests reported by the last four companies noted.)

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[1] See pp. 85, 89.

In making the statistical summary of this large number of tests the principles, elaborated in the "A.S.T.M. Manual on Presentation of Data," March, 1941, reprint, were used. The basic measures chosen for this study were: frequency distribution, computation of an average, and standard deviation.

While the actual frequency distribution is not shown in the tables, the number of tests employed in making such a distribution is represented. The average for each distribution is shown in the third column as symbolized by $\bar{X}$. The standard deviation, σ, is shown in the fourth column. The fifth column, computed by subtracting three times standard deviation (3σ) from the average $\bar{X}$, represents the probable minimum from any group of elongation values within the limits of wall thickness shown. Expecting this minimum, column 6 represents the percentage of the number of tests that would be expected to fail to meet the minimum elongation requirement of 35 per cent prescribed by the specifications.

Tables I and II, in addition to illustrating the significant effect that wall thickness has on the percentage of elongation obtained with a strip tension test specimen $1\frac{1}{2}$ in. in width and 2 in. in gage length, taken from various wall thicknesses of pipe manufactured to Specifications A 53 and A 106, also demonstrate the discrepancy in values resulting from use of the standard round tension test specimen with 2-in. gage length as compared to a strip specimen $1\frac{1}{2}$ in. in width in the heavier wall sections—0.750 in. and over—where the round specimen is now mandatory. It should be noted that the cross-sectional area of the standard round specimen with diameter of 0.505 in. is the same as the cross-sectional area of a $1\frac{1}{2}$-in. strip specimen cut from pipe 0.134 in. in wall thickness.

Conclusions:

The following conclusions can be drawn from a study of Tables I and II:

1. It is practically impossible to meet a minimum elongation requirement of 35 per cent in grade A pipe when the wall thickness is lighter than $\frac{5}{16}$ in. (0.3125 in.). If an order were received for 2-in. standard pipe, the pipe tables call for 0.154 in. wall thickness. From Table I, or II it is shown that this wall thickness, which is within the limits of 0.134 to 0.201 in. would result in an average elongation of 37.8 per cent, while the probable minimum would be as low as 28.1 per cent. From a normal conception of an average value, it follows that about half of the tests would fall below 38.7 per cent. Since 35 per cent is the specification minimum there are certain percentages of the pipe with elongation values between 35 and 38 per cent which will meet the specifications. To determine this statistical number, subtract from 50 per cent to obtain the percentage of tests that will not meet the required elongation of 35 per cent which results, in this case, in a figure of 18.5 per cent. It will be noted that in the case of thinner walls the percentage of tests not meeting the prescribed minimum is very high.

2. Again referring to Table I, it is shown that the minimum elongation

TABLE I.—STATISTICAL SUMMARY OF ELONGATION VALUES OF GRADE A PIPE SHOWING EFFECT OF WALL THICKNESS.

Chemical Composition:	
Carbon, per cent	0.08 to 0.20
Manganese, per cent	0.30 to 0.50

Tensile Requirements:	
Tensile strength, min., psi	48 000
Yield point, min., psi	30 000
Elongation in 2 in., min., per cent	35

Wall Thickness, in.	Number of Tests	Average Elongation, per cent (X)	Standard Deviation, per cent (σ)	Probable Minimum, per cent $(\bar{X} = 3\sigma)$	Percentage of Tests not Meeting Minimum Elongation of 35 per cent	Proposed Values (35 per cent − 1.75 per cent for each $\frac{1}{32}$ in. below 0.312 in. wall thickness)		
						Wall Thickness, in.	Elongation, per cent	Percentage of Tests not Meeting the Proposals
0.100 and under	186	28.5	3.4	18.3	97.0	0.062 0.093	21.00 22.75	1.40 ....
0.100 to 0.134	122	32.7	3.1	23.4	77.0	0.124	24.50	....
0.134 to 0.201	1016	37.8	3.1	28.1	18.5	0.156 0.187	26.25 28.00	.... 0.08
0.201 to 0.268	893	41.2	2.8	32.8	1.3	0.218 0.250	29.75 31.50	 0
0.268 to 0.335	906	42.7	2.4	35.5	0	0.280 0.312	33.25 35.00	 0
0.335 to 0.402	404	43.4	2.9	34.4	0		35.00	0
0.402 to 0.469	76	44.8	3.1	35.5	0		35.00	0
0.469 to 0.536	138	44.2	3.0	35.2	0		35.00	0
0.536 to 0.603	91	44.7	3.4	34.5	0		35.00	0
0.670 to 1.075	150	45.9	3.3	36.0	0		35.00	0
0.505[a]	25	36.3	3.7	25.2	36.3		28	

[a] Round 2-in. gage length specimen.

TABLE II.—STATISTICAL SUMMARY OF ELONGATION VALUES OF GRADE B PIPE SHOWING EFFECT OF WALL THICKNESS.

Chemical Composition:	
Carbon, per cent	0.17 to 0.30
Manganese, per cent	0.50 to 0.80

Tensile Requirements:	
Tensile strength, min., psi	62 000
Yield point, min., psi	35 000
Elongation in 2 in., min., per cent	25

Wall Thickness, in.	Number of Tests	Average Elongation, per cent $(\bar{X})$	Standard Deviation, per cent (σ)	Probable Minimum, per cent $(\bar{X} = 3\sigma)$	Percentage of Tests Not Meeting Minimum Elongation of 25 per cent	Proposed Values (30 per cent − 1.50 per cent for each $\frac{1}{32}$ in. below 0.312 in. wall thickness	
						Wall Thickness, in.	Elongation, per cent
0.100 and under	110	27.9	2.8	19.5	15.0	0.062 0.093	18.00 19.50
0.100 to 0.134	208	29.9	3.5	19.4	8.0	0.124	21.00
0.134 to 0.201	463	33.9	2.6	26.1	0	0.156 0.187	22.50 24.00
0.201 to 0.268	904	37.3	2.6	29.5	0	0.218 0.250	25.50 27.00
0.268 to 0.335	1126	37.9	2.5	30.4	0	0.280 0.312	28.50 30.00
0.335 to 0.402	350	39.7	2.4	32.5	0		30.00
0.402 to 0.469	28	39.9	2.8	31.5	0		30.00
0.469 to 0.536	112	39.3	2.5	31.8	0		30.00
0.536 to 0.603	52	38.8	1.9	33.1	0		30.00
0.670 to 1.075	180	38.7	3.6	27.9	0		30.00
0.505[a]	13	29.8	3.1	20.5	6.2		22.00

[a] Round 2-in. gage length specimen.

value of 35 per cent as now prescribed in these specifications is statistically the proper minimum and should not be raised above this figure. In standard or extra-heavy wall thicknesses, as, for example, 6-in. standard pipe, which calls for a wall thickness of 0.280 in., it is shown that while an average of 42.7 per cent would be obtained, a minimum of 35.5 per cent must be expected, and any increase of this minimum would result in a large rejection of material. This indicates that the existing elongation requirement was based on properties of common pipe sizes and was undoubtedly proper at the time these specifications were written.

3. An analysis of grade B tests in Table II shows that the effect is not so pronounced as it is with grade A. The minimum elongation value for grade B prescribed in the specifications is 25 per cent in 2-in. gage length as compared with 35 per cent for grade A. The results do indicate, however, that serious trouble can be expected in the lighter walls.

4. The mandatory use of a standard round 2-in. gage length test specimen has a very definite effect on the elongation value obtained as compared with a strip tension test specimen $1\frac{1}{2}$ in. in width from the same wall thickness. For grade A the minimum to be expected is 25 per cent, and for grade B, 20.5 per cent, which would result in the rejection of 36.3 per cent of tests for grade A and 6.2 per cent for grade B. Comparing this with a strip test specimen from comparable wall thicknesses, it is shown from results that in both analyses all strip tests would meet the suggested minimum.

5. No trouble can be expected in meeting the minimum elongation values of the specifications, using a strip tension test specimen $1\frac{1}{2}$ in. in width in wall thicknesses heavier than $\frac{5}{16}$ in. (0.3125 in.). But it is significant that the minimum elongation value to be expected in these wall thicknesses borders on the minimum now prescribed. This is particularly true with grade A.

Summary and Recommendations:

This statistical analysis demonstrates that the effect of wall thickness on the percentage of elongation value resulting from tension testing of light wall pipe or tubing is very pronounced. It also demonstrates that the results from the standard round 2-in. gage length specimens should not be compared with the results obtained from a strip test specimen $1\frac{1}{2}$ in. in width in comparable wall thicknesses. It seems a reasonable conclusion to state that it is virtually impossible to meet the minimum elongation requirements of the specifications discussed.

It will be noted from the section of the main report covering Pipe and Tubular Products[2] that revisions are proposed which, without altering the basic elongation minimum as now prescribed for pipe or tubular products, would seem to offer an equitable adjustment.

These changes are summarized as follows:

(*1*) If the specified wall thickness is lighter than $\frac{5}{16}$ in. (0.3125 in.), the minimum elongation shall be computed by subtracting 5 per cent of the specifications basic minimum (1.75 per cent for grade A and 1.50 per cent for grade B in Specifications A 53 and A 106, longitudinal) for each $\frac{1}{32}$ in. of the specified wall thickness below $\frac{5}{16}$ in.

(*2*) If the wall thickness is $\frac{3}{4}$ in. or over, and the standard round 2-in. gage length specimen is used, then the minimum elongation would be about 75 to 80 per cent of the basic minimum of the specifications.

(*3*) As an alternate to item (*2*) above, the strip test specimen $1\frac{1}{2}$ in. in width may be used without reducing the basic minimum of the specifications (35 per cent).

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[2] See p. 84.

That these are not drastic reductions which would place the quality of the material from the standpoint of ductility in jeopardy is shown in the last three columns of Tables I and II. It should be observed that the values shown are absolute minima (not average) and by the same principles used in the statistical analysis of the original data, it can be shown that the actual values would be higher than the proposed minima to a considerable degree. For example, if a minimum value of 21 per cent is required, and we assume a standard deviation value of 3.4 per cent, then the average value would approximate 3 times 3.4 per cent plus 21 or 31.2 per cent. Mathematically, with the normal conception of an average, 50 per cent of the tests would exceed 31.2 per cent, or actually test as high as 41.2 per cent. It is apparent that the range would be from 21 to 41 per cent.

APPENDIX II

STATEMENT ON FLATTENING TEST REQUIREMENTS FOR STEEL PIPE

Committee A-1 on Steel, through its Subcommittees IX on Steel Tubing and Pipe and XXII on Valves, Fittings, Piping and Flanges for High-Temperature and Subatmospheric-Temperature Service, is recommending revisions in the flattening test requirements in the Standard Specifications for Welded and Seamless Steel Pipe (A 53 - 40) and for Lap-Welded and Seamless Steel Pipe for High-Temperature Service (A 106 - 41), and in the Tentative Specifications for Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F. (A 158 - 41 T) and for Seamless Carbon-Molybdenum Alloy-Steel Pipe for Service at Temperatures from 750 to 1000 F. (A 206 - 41 T).

These recommendations, as detailed in the main report,[1] are based on extensive studies conducted by a special committee consisting of members from both of the subcommittees. To have on record some of the essential information developed by the committee, including the derivation of the formula being recommended and also a comparison of existing flattening test requirements as compared with those which will become effective as the recommendations are eventually approved, this statement is appended to the report.

Discussion:

With a great number of sizes and extremely diverse wall thicknesses of pipe covered in standard specifications, it has become increasingly apparent that some more rational system of determining the amount of flattening that a pipe should withstand would be desirable. At the same time if agreement could be reached it might satisfy certain consumer groups which feel that more flattening should be done on heavy wall pipe; and objections of manufacturers to the rather drastic amount of flattening on thin wall material might be overcome.

The present requirements in some of the specifications listed above require the pipe to be flattened between parallel plates to an amount which may be one half or one third the outside diameter or 5 or 7 times the wall thickness. The first requirement decreases the severity as the diameter increases, while a requirement based on the constant factor times the wall thickness results in increased severity with larger diameter. It was felt that some logical series of increments might be developed if the diameter and wall thickness were both taken into consideration and the committee then investigated a formula, first developed in 1931, which was based on the idea of establishing a test requiring equal fiber elongation in flattening on all sizes of pipe, with provision for suitable variation for different grades of steel and different ratios of thickness to diameter.

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[1] See pp. 84 to 92.

Derivation of Flattening Test Formula:

$$S = \frac{(1 + e)t}{e + t/D}$$

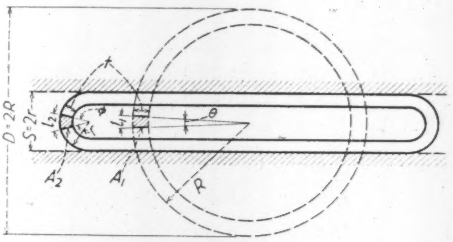
where:

S = distance between flattening plates, in inches,

t = nominal wall thickness of pipe, in inches,

D = actual outside diameter of pipe, in inches and

e = deformation per unit length (constant for a given grade of steel, 0.09 for grade A and 0.07 for grade B).



D = actual outside diameter of pipe
t = nominal wall thickness of pipe wall, in inches,
S = $2r$ = distance between plates after flattening,
R = $D/2$ = radius of tube before flattening,
l_1 = length of elementary arc,
l_2 = length of arc after test,
θ = angle subtended by l_1,
ϕ = angle subtended by l_2,
A_1 = area bounded by inside and outside diameter of tube and radial lines forming angle θ, and
A_2 = area bounded by inside and outside diameter of semicircular section of flattened specimen and radial lines forming angle ϕ.

FIG. 1.—Diagram Accompanying Flattening Test Formula.

Referring to Fig. 1, it is assumed that the position of the neutral line (point midway between the inside and outside surface of tube wall) remains fixed during the test; also that the curved portion of the test specimen remains circular in form. These assumptions, while not strictly true, are considered sufficiently accurate as to not affect the practical value of the results. The length of the test specimen should also be a few times the wall thickness to avoid the "end effect" due to bulging of the ends.

The change in length of the elementary arc at the exterior surface due to bending is derived as follows:

$$e = \frac{l_2 - l_1}{l_1} = \frac{r\phi - R\theta}{R\theta} \qquad (1)$$

and

$$A_1 = A_2 \qquad (2)$$

where:

$$A_1 = \int_{R-t}^{R} \int_{\theta}^{\theta} rdrde$$

which integrated gives:

$$A_1 = \frac{\theta}{2}(2Rt - t^2) \qquad (3)$$

Similarly

$$A_2 = \frac{\phi}{2}(2rt - t^2) \qquad (4)$$

Equating Eqs. 3 and 4:

$$\phi = \frac{(2R - t)}{(2r - t)}\theta \qquad (5)$$

Substituting in Eq. 1:

$$e = \frac{r\frac{(2R - t)}{(2r - t)}\theta - R\theta}{R\theta} \qquad (6)$$

Or

$$e = \frac{r\frac{(2R - t)}{(2r - t)} - R}{R}$$

From Fig. 1: $r = S/2$
$R = D/2$

Then

$$e = \frac{S/2\frac{(D - t)}{(S - t)} - \frac{D}{2}}{\frac{D}{2}}$$

$$eD + D = \frac{S(D - t)}{S - t}$$

$$\frac{e + 1}{S} = \frac{1 - t/D}{S - t}$$

$$S = \frac{S(e + 1) - t(e - 1)}{1 - t/D}$$

Dividing by S

$$1 = \frac{(e + 1) - t/s(e - 1)}{1 - t/D}$$

Multiplying by $(1 - t/D)$

$$1 - t/D = (e + 1) - t\,\frac{(1 + e)}{S}$$

$$S = \frac{(1 + e)t}{e + t/D}$$

$$\text{or } S = CD \text{ where } C = \frac{(1 + e)t/D}{e + t/D}$$

Results Using Formula and Comparison with Existing Requirements:

After agreeing on the mathematical analysis which is based upon the geometry of flattening to be used, the next step was to determine by study of numerous flattening test data the approximate fiber elongation factor "e" which might logically be used with the formula to cover various grades, sizes, and wall thicknesses of pipe.

Based on considerable data which

TABLE I.—COMPARISON OF PRESENT AND PROPOSED FLATTENING TEST REQUIREMENTS.

Size, in.	Nominal Wall Thickness, in.	Weight per Foot, Plain Ends, lbs.	Ratio, t/D	Distance Between Plates, in., Required by A.S.T.M. Specifications			Proposed Distance Between Plates, in., by Formula: $S = \frac{(1+e)t}{e+t/D}$		
				A 106		A 206	A 106		A 206, Carbon Molybdenum, $e = 0.08$
				Grade A	Grade B		Grade A, $e = 0.09$	Grade B, $e = 0.07$	
3	0.216	7.57	0.062	1.167	1.750	1.750	1.549	1.751	1.640
3	0.300	10.25	0.085	1.500	2.100	2.100	1.869	2.071	1.963
3	0.437	14.31	0.124	2.185	3.059	3.059	2.226	2.410	2.313
4	0.237	10.79	0.053	1.500	2.250	2.250	1.806	2.067	1.924
4	0.337	14.98	0.074	1.685	2.359	2.359	2.240	2.504	2.363
4	0.437	18.96	0.097	2.185	3.059	3.059	2.547	2.800	2.666
4	0.531	22.51	0.118	2.655	3.717	3.717	2.783	3.022	2.893
8	0.250	22.36	0.029	2.875	4.313	4.313	2.290	2.702	2.477
8	0.500	43.38	0.058	2.875	4.313	4.313	3.682	4.180	3.913
8	0.906	74.69	0.105	4.530	6.343	6.343	5.064	5.540	5.286
10	0.250	28.04	0.023	3.583	5.375	5.375	2.412	2.876	2.621
10	0.500	54.74	0.047	3.583	5.375	5.375	3.978	4.573	4.252
10	1.125	115.65	0.104	5.625	7.875	7.875	6.321	6.918	6.603
12	0.250	33.38	0.020	4.250	6.375	6.375	2.477	2.972	2.700
12	0.562	73.16	0.044	4.250	6.375	6.375	4.571	5.275	4.895
12	1.312	160.27	0.102	6.560	9.184	9.184	7.448	8.162	7.785
14	0.375	54.57	0.027	4.667	7.000	7.000	3.494	4.137	3.785
14	0.593	84.91	0.042	4.667	7.000	7.000	4.897	5.665	5.250
14	1.406	189.12	0.100	7.030	9.842	9.842	8.066	8.850	8.433
16	0.375	62.58	0.023	5.333	8.000	8.000	3.617	4.315	3.932
16	0.843	136.46	0.053	5.333	8.000	8.000	6.426	7.333	6.842
16	1.562	240.86	0.097	7.810	10.934	10.934	9.105	10.008	9.531
18	0.437	81.97	0.024	6.000	9.000	9.000	4.178	4.974	4.538
18	0.937	170.84	0.052	6.000	9.000	9.000	7.192	8.218	7.666
18	1.750	303.72	0.097	8.750	12.250	12.250	10.200	11.213	10.677
20	0.500	104.13	0.025	6.667	10.000	10.000	4.739	5.632	5.143
20	1.031	208.87	0.052	6.667	10.000	10.000	7.914	9.042	8.436
20	1.750	341.10	0.088	8.750	12.250	12.250	10.716	11.851	11.250
24	0.562	140.68	0.023	8.000	12.000	12.000	5.421	6.466	5.893
24	1.218	296.36	0.051	8.000	12.000	12.000	9.416	10.771	10.038
24	1.750	415.86	0.073	8.750	12.250	12.250	11.702	13.094	12.353

have been developed by the manufacturers the committee has established the following values of "*e*" for the specifications indicated:

Specifications		Value of *e*
A 53 and A 106	Grade A........	0.09
	Grade B........	0.07
A 158.........	Ferritic..........	0.08
	Austenitic	0.09
A 206..........................		0.08

In a report submitted to the members of Subcommittees IX and XXII, the Special Committee on Flattening Test Requirements included extensive tabular data showing the distance between the plates according to the present requirements and the proposed distance resulting from the use of this formula. To provide some idea of the changes involved, selections of the data are shown in the accompanying Table I.

From these data it will be readily apparent that for grade A pipe the formula does not generally require such rigorous flattening as is now the case, although in thinner walls the requirements according to the formula are more severe. In general, for grade B pipe and the carbon-molybdenum grade covered in Specification A 206, the values required by the formula in the sizes which would be most likely used for power piping and pressure work would be comparable or generally more severe than now required.

The recommendations setting up the use of this formula for the seamless grades A and B pipe covered in Specifications A 53 and in the other three specifications have been approved by letter ballot vote in Subcommittees IX and XXII.

REPORT OF SECTIONAL COMMITTEE
ON
STANDARDIZATION OF DIMENSIONS AND MATERIALS OF WROUGHT-IRON AND WROUGHT-STEEL PIPE AND TUBING

ASA PROJECT: B36

Sectional Committee B36 on Standardization of Dimensions and Materials of Wrought-Iron and Wrought-Steel Pipe and Tubing has not met during the year. However, a number of matters were handled by correspondence and as indicated, on the recommendation of the sectional committee, the American Standards Association reapproved two American Standards in which revisions had been made and likewise approved seven A.S.T.M. standards as American Standard for the first time assigning the following designations:

A.S.T.M. Standard Specifications for:

- Welded and Seamless Steel Pipe (A 53 – 40; ASA B36.1-1940),
- Lap-Welded and Seamless Steel Pipe for High-Temperature Service (A 106 – 41; B36.3-1942),
- Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes (A 83 – 40; B36.12-1942),
- Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 – 40; B36.13-1942),
- Seamless Steel Boiler Tubes for High-Pressure Service (A 192 – 40; B36.14-1942),
- Medium-Carbon Seamless Steel Boiler and Superheater Tubes (A 210 – 40; B36.15-1942),
- Spiral-Welded Steel or Iron Pipe (A 211 – 40; B36.16-1942),
- Seamless Alloy-Steel Boiler and Superheater Tubes (A 213 – 40; B36.17-1942), and
- Electric-Resistance-Welded Steel Boiler and Superheater Tubes for High-Pressure Service (A 226 – 40; B36.18-1942).

Since certain revisions are being submitted by A.S.T.M. Committee A-1 on Steel at this annual meeting in the following standards, Sectional Committee B36 will give consideration during the year to the advisability of submitting these standards to the ASA for reapproval as American Standard in their revised form:

A.S.T.M. Standard Specifications for:

- Welded and Seamless Steel Pipe (A 53 – 40; ASA B36.1-1940),
- Lap-Welded and Seamless Steel Pipe for High-Temperature Service (A 106 – 41; B36.3-1942),
- Electric-Fusion-Welded Steel Pipe (Sizes 30 in. and Over) (A 134 – 39; B36.4-1939),
- Electric-Resistance-Welded Steel Pipe (A 135 – 34; B36.5-1935),
- Electric-Fusion Welded Steel Pipe (Sizes 8 in. to but not Including 30 in.) (A 139 – 39; B36.9-1939),
- Electric-Fusion-Welded Steel Pipe for High-Temperature and High-Pressure Service (A 155 – 36; B36.11-1939),
- Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes (A 83 – 40; B36.12-1942), and Seamless Alloy-Steel Boiler and Superheater Tubes (A 213 – 40; B36.17-1942).

Respectfully submitted on behalf of the sectional committee,

H. H. MORGAN,
Chairman.

SABIN CROCKER,
Secretary.

REPORT OF COMMITTEE A-2

ON

WROUGHT IRON

While Committee A-2 on Wrought Iron has held no meeting during the past year, the committee has furthered some of its activities by correspondence, particularly as regards the status of certain standard and tentative specifications.

One resignation has been recorded since the last annual meeting; the membership of the committee now totals 52, of whom 17 are classified as producers, 28 as consumers, and 7 as general interest members.

I. Adoption of Tentative Standard as Standard

Since no adverse criticisms have been received, the committee recommends that the Tentative Specifications for Single and Double Refined Wrought-Iron Bars (A 189 – 39 T) be approved without change for reference to letter ballot of the Society for adoption as standard.

II. Reaffirmation of Standards

The committee has reviewed the following specifications and definitions, which have been in effect for six or more years without revision, and since they are considered to be in agreement with current practice, they have been reaffirmed by the committee by letter ballot vote:

Standard Specifications for:
Common Iron Bars (A 85 – 36),
Refined Iron Bars (A 41 – 36),
Riveted Steel and Wrought-Iron Pipe (A 138 – 34), and
Standard Definitions of:
Terms Relating to Wrought Iron (A 81 – 33).

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 52 members; 47 members returned their ballots, of whom 41 have voted affirmatively, 0 negatively, and 6 members marked their ballots "not voting."

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, C. B. Bryant.
Vice-Chairman, James Aston.
Secretary, W. C. Masters.

This report has been submitted to letter ballot of the committee which consists of 52 members; 44 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

C. B. Bryant,
Chairman.

W. C. Masters,
Secretary.

REPORT OF COMMITTEE A-3

ON

CAST IRON

Since the 1941 annual meeting of the Society in Chicago, Ill., Committee A-3 on Cast Iron has held one meeting, in Cleveland, Ohio, on April 23, 1942. Another meeting will be held in June during the annual meeting of the Society.

It is with sincere regret that the committee records the death of Harold J. Stein of the Allis-Chalmers Mfg. Co.

The following resolution has been adopted by the committee:

> RESOLVED: That in the death of Harold J. Stein, who was director of research, chemistry, and metallurgy with the Allis-Chalmers Manufacturing Co., Milwaukee, Wis., as well as a member of Committee A-3 and other committees of the A.S.T.M., this committee recognizes and regrets the loss of his services.

Since the 1941 annual meeting one new member has been elected, C. H. Lorig, Battelle Memorial Institute. The resignation of F. K. Vial was accepted with regret.

The following changes in membership are recorded.

E. K. Smith, now representing the Ordnance Dept. (formerly Electrometallurgical Corp.),
A. J. Edgar, now representing the Gray Iron Institute, and
John T. Jarman now representing the Allis-Chalmers Mfg. Co.

The total membership of the committee is now 112, of whom 35 are classified as producers, 48 as consumers, and 29 as general interest members.

In accordance with the instructions of the committee, cooperation has been extended to the American Foundrymen's Association in its efforts in promoting understanding and utilization of A.S.T.M. specifications.

A War Advisory Board has been appointed as instructed by the committee. This Board is prepared to help and advise in all practical ways in matters of specification and technology which may be referred to them by Governmental agencies through the medium of A.S.T.M. Headquarters. The appointees are:

J. T. MacKenzie (chairman),
W. L. Collins,
R. G. McElwee,
G. P. Phillips,
W. H. Rother, and
J. W. Bolton and E. R. Young, *Ex-officio*.

As is evident in the following recommendations and subcommittee reports, work in clarification and simplication has been directed toward removal of obselete specifications, and the directing of other specifications toward the General Castings Specifications A 48 – 41 which are considered suitable as master specifications, having value for selection as well as for procurement whenever definite engineering properties are desired. Federal Specification QQ-I-652 follows the strength and section provisions of Specifications A 48. In the opinion of the committee, coming substitutions of gray iron to replace more critical metals should be specified under true engineering specifications such as A.S.T.M. Standard A 48 and Federal QQ-I-652, for all specific applications. (Distinction should be made between product specifications and material specifications.)

EMERGENCY ALTERNATE PROVISIONS

On the recommendation of Committee A-3, proposed Emergency Alternate Provisions in the Standard Specifications for Lightweight and Thin-Sectioned Gray-Iron Castings (A 190 - 40), which provide for the deletion of certain references to physical properties, were accepted by Committee E-10 on Standards on June 18, 1942 and have been published as stickers for attachment to the specifications to which they apply. They have also been published in the ASTM BULLETIN, No. 117, August, 1942, p. 73. This emergency alternate is believed desirable since the basic objective of the specifications is to promote production of light sectioned castings of maximum machinability and the meeting of minimum physical requirements is contrary to this objective, and likely to cause misunderstanding and loss of time and tools in war production.

I. NEW TENTATIVE STANDARD

The committee recommends that the Method of Compression Testing of Cast Iron,[1] be accepted for publication as tentative.

II. REVISION OF TENTATIVE STANDARD

On the recommendation of Subcommittee X on Nomenclature and Definitions, the committee recommends that the Tentative Definitions of Terms Relating to Cast Iron (A 196 - 36 T) be revised as appended hereto[2] and continued as tentative.

III. REVISION OF STANDARD, IMMEDIATE ADOPTION

On the recommendation of Subcommittee XVII on Gray Iron Castings for Valves, Flanges, and Pipe Fittings, the committee recommends for immediate adoption the following revisions in the Standard Specifications for Gray Iron Castings for Valves, Flanges, and Pipe Fittings (A 126 - 40),[3] and accordingly asks for a nine-tenths affirmative vote at the annual meeting in order that these modifications may be referred to letter ballot of the Society. This action is subject to concurrence by the Manufacturers Standardization Society.

Section 2.—Change to read as follows by the addition of the italicized words and the omission of the words in brackets:

> 2. [Process] *Control.*—[The iron shall be made by the cupola, air-furnace, electric-furnace, or other approved process.] The iron shall be produced under regular chemical and physical control [and shall be gray iron of high quality] *in accordance with these specifications.*

Section 7.—Make this section paragraph (*a*) and change the first sentence to read as follows by the addition of the italicized words:

> Tension test specimens, *which may be* cast as shown in Fig. 1, shall conform to the dimensions shown in Fig. 2, shall be reasonably smooth and round at breaking section, and shall be tested without machining except that the enlarged ends shall be threaded (1¾-in. U. S. standard threads are recommended).

Add a new paragraph (*b*) to read as follows:

> (*b*) Test specimens described herein have been developed to maintain uniformity of product control, particularly for large scale production of articles covered in Section 1 on scope. However, these specifications extend to the founder, manufacturer, or producer the option, either for convenience in testing materials on individual jobs or orders placed with manufacturers not regularly producing castings under the scope of these specifications, or as a regular practice, to substitute test specimens and testing procedure in accordance with the Standard Specifications for Gray Iron Castings (A.S.T.M. Designation: A 48) of the American Society for Testing Materials. In cases of such substitutions, the requirements of Standard A 48 for both tensile (as determined by the B or 1.20 in. diameter bar) and transverse tests and these requirements only shall govern and classes 20, 30, and 40 of Standard A 48 shall be considered equivalent to classes A, B, and C, respectively, of Specifications A 126.

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[1] This method was accepted as tentative by the Society and appears in the 1942 Book of A.S.T.M. Standards, Part I, p. 1077.

[2] These revised definitions were accepted as tentative by the Society and appear in the 1942 Book of A.S.T.M. Standards, Part I, p. 1090.

[3] 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 140.

Figure 1.—Add an asterisk next to the dimensions shown on the ingress gate referring to a note reading as follows:

NOTE.—These dimensions are suggested as satisfactory for average conditions, but may be varied best to suit individual pouring practices.

IV. ADOPTION OF TENTATIVE STANDARD AS STANDARD

The committee recommends that the Tentative Specifications for Cast Iron Soil Pipe and Fittings (A 74 – 39 T)[4] be approved without change for reference to letter ballot of the Society for adoption as standard.

V. WITHDRAWAL OF STANDARD AND TENTATIVE STANDARD

It is recommended that the Standard Specifications for Cast-Iron Locomotive Cylinders (A 45 – 14)[5] and the Tentative Specifications for Chilled-Tread Cast-Iron Wheels (A 46 – 30 T)[5] be withdrawn in accordance with action taken at the June, 1941, and March, 1942, meetings, respectively.

VI. EDITORIAL CHANGES IN STANDARD AND TENTATIVE STANDARD

The following editorial changes in the Tentative Recommended Practice for Evaluating the Microstructure of Graphite in Gray Iron (A 247 – 41 T)[6] were recommended by Subcommittee VII on Microstructure of Cast Iron and approved by Committee A-3 at its Cleveland meeting:

Appendix.—Change the title of the appendix from "Recommended Procedure for Polishing Microsections of Cast Iron," to read "Suggested Procedures for Polishing Microsections of Cast Iron."

Add as an alternative procedure the method of Beregekoff and Forgeng.[7]

The following editorial change in the Standard Specifications for Gray Iron Castings (A 48 – 41)[8] was recommended jointly by Subcommittee VI on General Castings and Subcommittee XI on Methods of Testing and approved by Committee A-3 at its Cleveland meeting:

Figure 2.—Change the machined diameter of the type B tension test specimen from "0.800" to read "0.750" in.

This change does not affect the strength test values and eliminates the tendency toward shoulder breaks sometimes encountered in testing higher strength classes of iron with reduced section 0.800 in. in diameter.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 112 members, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARD			
Compression Testing of Cast Iron	60	0	10
II. REVISION OF TENTATIVE STANDARD			
Def. of Terms Relating to Cast Iron (A 196 – 36 T)	71	0	3
III. REVISION OF STANDARD, IMMEDIATE ADOPTION			
Spec. for Gray Iron Castings for Valves, Flanges, and Pipe Fittings (A 126 – 40)	59	0	10
IV. ADOPTION OF TENTATIVE STANDARD AS STANDARD			
Spec. for Cast Iron Soil Pipe and Fittings (A 74 – 39 T)	57	0	13
V. WITHDRAWAL OF STANDARD AND TENTATIVE STANDARD			
Spec. for Cast-Iron Locomotive Cylinders (A 45 – 14)	49	3	3
Spec. for Chilled-Tread Cast-Iron Wheels (A 46 – 30 T)	56	0	16

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Pig Iron (H. M. Rich, chairman).—A revision of the

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[4] 1939 Book of A.S.T.M. Standards, Part I, p. 974.

[5] *Ibid.*, pp. 491 and 976.

[6] 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 300.

[7] This new procedure has been incorporated in the Tentative Recommended Practice A 247 which appears in its latest form in the 1942 Book of A.S.T.M. Standards, Part I, p. 1079.

[8] 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 95.

Standard Specifications for Foundry Pig Iron (A 43 – 24) has been prepared, and submitted to letter ballot in the subcommittee. This revision will be presented to Committee A-3 at its meeting in June.

Subcommittee III on Cast Iron Locomotive Cylinders.—The resignation of O. B. Schultz as chairman has been accepted with regret and the subcommittee disbanded. The withdrawal of the Standard Specifications for Cast-Iron Locomotive Cylinders (A 45 – 14) has been recommended as mentioned earlier in this report.

Subcommittee IV on Cast-Iron Car Wheels.—The resignation of F. K. Vial as chairman has been accepted with regret and the subcommittee disbanded. The withdrawal of the Tentative Specifications for Chilled-Tread Cast-Iron Wheels (A 46 – 30 T), originally prepared by this subcommittee, is being recommended as mentioned earlier in this report.

Subcommittee VI on General Castings (H. Bornstein, chairman).—This subcommittee recommends to Committee A-3 that Standard A 48 should be considered the master specifications for cast iron, and that other specifications of the Society, involving gray iron castings wherein strength of the material is a consideration, conform in general with Specifications A 48.

Subcommittee VII on Microstructure of Cast Iron (G. F. Comstock, chairman).—An alternate polishing method is recommended as an addition to suggested polishing methods in the Tentative Recommended Practice for Evaluating the Microstructure of Graphite in Gray Iron (A 247 – 41 T).

Studies of methods adaptable to revealing cell size in gray iron will be undertaken. This is and has been recognized as a significant structural feature, and research to establish definite and improved techniques for its revelation is contemplated.

Subcommittee VIII on Cast-Iron Culvert Pipe (W. J. Schlick, chairman).—It is reported that culvert pipe is not a current item of manufacture during the period of National Emergency.

Subcommittee X on Nomenclature and Definitions (A. J. Herzig, chairman).—As mentioned earlier in this report, this subcommittee has recommended revisions in the Tentative Definitions of Terms Relating to Cast Iron (A 196–36 T).

Subcommittee XI on Methods of Testing (V. A. Crosby, chairman).—By letter ballot, this subcommittee recommends that Standard A 48 – 41, be considered the master specifications for gray iron castings of the Society and that the test bars and tensile strength and transverse strength requirements thereof be adopted as the basis for all specifications under the jurisdiction of Committee A-3 wherein the strength characteristics of the material are a consideration.

A proposed Method of Compression Testing of Cast Iron[1] has been prepared by this subcommittee, and is presented for publication as tentative. A paper on "Compression Testing of Cast Iron," by W. Leighton Collins is appended to this report.

A recommended practice on torsion testing has been prepared and, if approved by letter ballot of the subcommittee, will be presented to Committee A-3 at the June meeting.[9]

Subcommittee XV on Impact Testing (J. T. MacKenzie, chairman).—Neither the subcommittee nor the testing machine manufacturers feel that work on development of a "standard" machine is justified at this time.

Subcommittee XVI on Heat Treatment of Cast Iron (J. S. Vanick, chairman).—Investigation of effects of changes in dimensions upon design or preferred tension test specimen and method of testing hardened unmachinable cast iron is being continued. A proposed Recommended Practice for Tension Testing of Heat-Treated Gray Iron Castings is in preparation by this subcommittee.

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[9] See Editorial Note, p. 110.

As representative of Committee A-3 on the E-8 Subcommittee on Definitions of Terms Relating to Heat Treatment, J. S. Vanick has cooperated in the review of the proposed Glossary of Heat Treating Terms prepared by the Joint Committee on Definitions of Terms Relating to Heat Treatment, consisting of representatives of the A.F.A., A.S.M., S.A.E., and A.S.T.M.[10]

Subcommittee XVII on Gray Iron Castings for Valves, Flanges, and Pipe Fittings (J. J. Kanter, chairman).—This subcommittee wishes to emphasize that the Standard Specifications for Gray Iron Castings for Valves, Flanges, and Pipe Fittings (A 126 – 40) are primarily product specifications and are not intended to include provisions for an engineering material in terms of strength in given sections. These specifications were prepared in response to a request from the Manufacturers Standardization Society of the Valve and Fittings Industry and have proved suitable for certification as to the relative quality of metal as poured into the test bar covered in the specifications. The revisions being recommended in these specifications, mentioned earlier in this report, are presented subject to approval by letter ballot of this subcommittee and to confirmation by the M.S.S.

Subcommittee XVIII on Automotive Castings (A. L. Boegehold, chairman).—Curtailment of production makes it unnecessary to give consideration to this product for domestic use at this time. The subcommittee awaits any requests that may be made with respect to similar products for war usage.

This report has been submitted to letter ballot of the committee, which consists of 112 members; 70 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. W. BOLTON,
Chairman.

E. R. YOUNG,
Secretary.

J. T. MACKENZIE,
Vice-Chairman.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee A-3 presented to the Society through Committee E-10 on Standards a new Recommended Practice for Torsion Tests of Cast Iron. This recommended practice was accepted by Committee E-10 for publication as tentative on August 24, 1942, and appears in the 1942 Book of A.S.T.M. Standards, Part I, p. 1087, bearing the A.S.T.M. serial designation A 260 – 42 T.

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[10] See p. 482.

APPENDIX

COMPRESSION TESTING OF CAST IRON

By W. Leighton Collins[1]

Synopsis

A useful and reliable method of compression testing that has been used satisfactorily at the University of Illinois and that can be used in any testing laboratory is presented. The types of specimens and the compression tool to secure axial loading are described.

The need for information on compression testing of cast iron results from an attempt of Subcommittee XI on Methods of Testing of the Society's Committee A-3 on Cast Iron to prepare a recommended practice for compression testing of cast iron. Because of the many questions that arose, such as method of loading and type of specimen, the author was asked to present a description of the apparatus used to insure axial loading, the type of specimen, and the test procedure that have been satisfactorily used at the University of Illinois. It is not intended that this should be an explanation of a highly precise and time-consuming research procedure but rather that it be a discussion of a useful and reliable method of careful testing that can be followed in any testing laboratory and that will give reproducible results which are real measures of the properties of cast iron and not values that are a function of the method of testing.

The apparatus used to insure a satisfactory degree of axiality of loading is shown in Fig. 1 and consists of a heavy, closed steel frame through the top of which a vertical plunger moves. The specimen to be tested is placed between the plunger and a hardened steel anvil which rests on the bottom of the frame. It will be noted that the device is similar to the compression tool shown in Fig. 3 of A.S.T.M. Tentative Methods of Compression Testing of Metallic Materials (E 9 – 33 T),[2] except that the frame is made more rigid by making it a closed "ring" in order to reduce to a minimum the bending of the specimen due to deformation of the frame. Several similar devices, open on one side and also with closed frames of cast iron, had been built and used but were discarded because they were not sufficiently rigid or broke too easily if the top bearing block was accidentally run down onto the frame.

Axiality of loading is produced by means of a ball bearing at the top of the plunger and a soft steel plate with a "Brinell impression," by having a close fit between plunger and frame and by having the horizontal surfaces of the bottom of the frame, the hardened anvil, and the plunger as nearly parallel as possible. The ends of the specimen, of course, must be perpendicular to the axis of the specimen and carefully machined; the specimen itself also must be carefully centered on the anvil by means of a split collar, as described in the next paragraph.

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[1] Assistant Professor, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[2] 1939 Book of A.S.T.M. Standards, Part I, p. 1230.

The ball bearing and soft-steel plate eliminate most of the undesirable effects resulting from improper alignment of the heads of the testing machine or rotation of the movable head as load is applied. The long guide for the plunger increases rigidity, and also insures that the face of the plunger moves vertically downward as load is applied; it also means that forces perpendicular to the plunger, and thus friction resisting the applied load, will be small. The hardened steel anvil is carefully centered in the bottom of the frame by means of a hole in the frame and a lug on the anvil. The anvil is also carefully turned in order that the specimen can be centered by means of a split collar, shown in Fig. 1. The collar, machined to fit the anvil and specimen, is used only to center the specimen and is removed during actual testing. A different collar is used for each size of specimen. It should also be noted that the bottom of the frame has a hole, concentric with the axis of the plunger and anvil, into which a close-fitting pin can be inserted for centering the entire apparatus on the weighing platform of the testing machine.

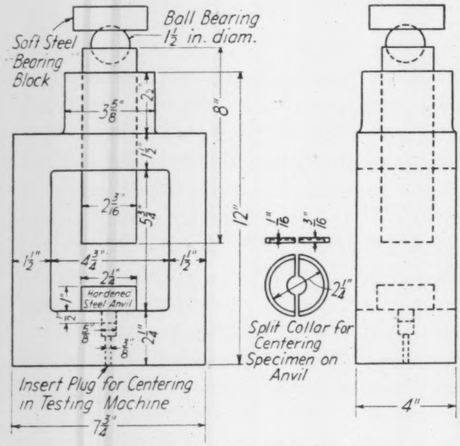
Soft Steel Bearing Block
Ball Bearing 1½ in. diam.
3⅝"
8"
2½"
1½"
2 3/16"
5¾"
12"
1½"
4¾"
1½"
2¼"
Hardened Steel Anvil
1/16"
3/16"
2¼"
Split Collar for Centering Specimen on Anvil
Insert Plug for Centering in Testing Machine
7¾"
4"

FIG. 1.—Compression Tool.

The precautions mentioned above have permitted the use of cylindrical specimens, that is, specimens not having the flared ends shown in Fig. 1 of Tentative Methods E 9 - 33 T.[2] No premature cracking or chipping of the ends of specimens has been observed; likewise, failures due to bending or fracture have not occurred consistently on a side toward any one given point of the apparatus. In fact, the apparatus has been used successfully for compression testing of both steel[3] and cast iron.[4]

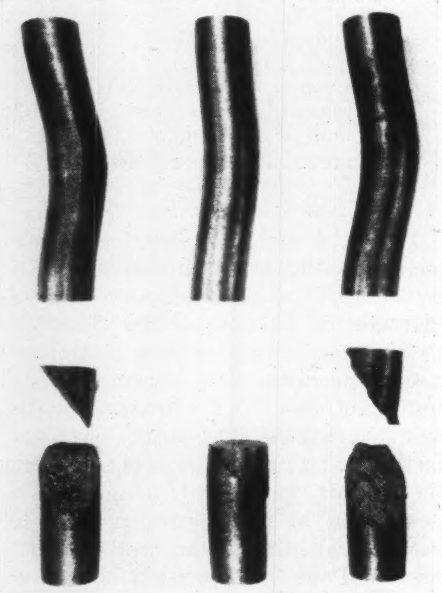

FIG. 2.—Ruptured Specimens of Cast Iron.

Top—$L/d = 6$

Bottom—$L/d = 3$.

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[3] W. Leighton Collins, Discussion of paper by Bruce Johnston and Francis Opila, "Compression and Tension Tests of Structural Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 574 (1941).

[4] W. Leighton Collins and James O. Smith, "The Notch Sensitivity of Alloyed Cast Irons Subjected to Repeated and Static Loads," see p. 639.

The specimens used by the author have not had flared ends because a specimen having a diameter of 0.75 in., a gage length of 2 in. (for determining modulus of elasticity) and a small length to diameter, $\frac{L}{d}$, ratio to avoid excessive bending due to column action cannot be obtained from a 1.2-in. diameter flexure test bar. For determining ultimate strength, specimens having a diameter of 0.75 in. and an $\frac{L}{d}$ ratio of 3 have been used. The apparatus shown in Fig. 1 permits the use of specimens having a diameter of 0.75 in. and an $\frac{L}{d}$ ratio of as great as 6, and such specimens have been used for determining modulus of elasticity for both steel[3] and cast iron. A photograph of ruptured specimens of cast iron of both sizes is shown in Fig. 2.

It appears to the author that the flared ends were designed to secure a distribution of stress throughout the length of constant diameter that is not greatly influenced by friction on the ends of the specimen and to approach more nearly fixed end conditions, to reduce bending, for specimens with large values of $\frac{L}{d}$. Whether specimens with flared ends would give the same results as specimens of the same diameter and with the same constant diameter length but without flared ends is a problem of importance and requires some experimental study. The author suggests that Committee A-3 undertake such an investigation.

DISCUSSION

MR. J. S. VANICK[1].—This paper by Mr. Collins constitutes the frame-work upon which Committee A-3 on Cast Iron built its recommended practice for compression testing.[2] There are several laboratories other than the University of Illinois that employ the straight end specimen. The committee encountered some criticism of the proposed method to the extent of pointing out that precision in squaring the ends of the specimen was necessary, and that spherical joints in the testing fixture were necessary to avoid edge cracking of the specimen under test, particularly with hard or brittle and possibly white iron. Since such irons are in the minority and testing them under compression is infrequent, it was considered possible that under such difficult conditions specimens with the bell-shaped ends might be necessary to increase the bearing area and reduce the risk of edge cracking on the ends. Therefore, Mr. Collins makes a plea in his paper that this point be kept under observation. Meanwhile, in the interest of simplicity and economy the straight end specimen was adopted in the committee's recommended practice.

MR. M. F. SAYRE.[3]—I am glad to see this evidence of additional interest in the compression testing of metals. A word or so, however, might be said about what I suspect is the reason for some of the peculiarities in compression testing. I rather think that the real reason for the use of the spherical seat rather than simple flat ends is the inaccuracy in machining, as of some years ago, and that with the increased accuracy of machine work now obtainable, the need for a spherical seat is decreasing. Along with that has come an improvement in technique of construction of the testing machines themselves.

A word of caution should be given as regards the compression fixture described here. The fixture contains a plunger working in a rather closely fitting sleeve, loaded by means of a firmly seated ball at the top. If the testing machine is in an ideal condition, with the fixture placed vertically in the machine, then the up-and-down movement of the testing machine will cause the plunger to move parallel to itself; but if there are any defects in the testing machine, then the head will tend to move sideways slightly and produce what may be quite a large friction between the plunger and the sleeve. This could probably be obviated by the use either of the ball bearing joint shown in the top of the compression fixture in the Tentative Methods of Compression Testing of Metallic Materials (E 9 – 33 T),[4] or by the use of some other device which will allow a certain amount of lateral play.

I do not think that this is a major objection, but I think it is an indication that caution must be used in operating with this particular type of fixture.

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[1] Metallurgist, The International Nickel Co., Inc., New York, N. Y.

[2] Tentative Method of Compression Testing of Cast Iron (A 256 – 42 T), 1942 Book of A.S.T.M. Standards, Part I, p. 1077.

[3] Professor of Applied Mechanics, Union College, Schenectady, N. Y.

[4] 1939 Book of A.S.T.M. Standards, Part I, p. 1230.

MR. H. F. MOORE.[5]—As Mr. Collins has kindly but erroneously given me credit for designing this compression fixture described in the paper, I wish first to deny that. It is copied quite closely, with modifications, from one proposed in this country by R. R. Moore. In the apparatus as he used it there were placed balls or rollers underneath the yoke on the table of the testing machine, so that some sideways motion could take place with very little friction. As a matter of fact, the device now used at the University of Illinois was designed for student use, and the rollers were found to be a great deal more trouble than they were worth and were left out. I think the idea came first from some of the British laboratories.

MR. W. LEIGHTON COLLINS[6] (*author's closure*).—Mr. Vanick's statement that "hard or brittle and possibly white iron" might crack along the edges if cylindrical specimens are used, is a worthy comment. The author's tests have not included such materials and consequently no data other than those already referred to,[7] in which ultimate compressive strength varied from 80,000 psi. to 236,000 psi., can be cited.

The author agrees with Mr. Sayre that the device described does not provide adequately for undesirable lateral shifting of the head of a defective testing machine. The ball-bearing joint shown at the top of the compression tool in the Tentative Methods E 9 – 33 T,[4] would, as Mr. Sayre states, permit such undesirable translatory motion without producing excessive friction. Also, as Mr. Moore states, balls or rollers placed under the yoke of the compression tool on the table of the testing machine is another method of overcoming the same difficulty.

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[5] Professor of Engineering Materials, University of Illinois, Urbana, Ill.

[6] Assistant Professor, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[7] W. Leighton Collins and James O. Smith, "The Notch Sensitivity of Alloyed Cast Irons Subjected to Repeated and Static Loads," see p. 639.

REPORT OF COMMITTEE A-5
ON
CORROSION OF IRON AND STEEL

Committee A-5 on Corrosion of Iron and Steel has held two meetings during the past year: one on June 24, 1941, during the annual meeting of the Society in Chicago, Ill., and the other in Philadelphia, Pa., on January 14, 1942.

Since the last report 6 new members have been elected and 6 members have resigned. The committee membership now totals 130, of whom 58 are classified as producers, 44 as consumers, and 28 as general interest members.

EMERGENCY STANDARD

Emergency Specifications for Lead Coating (Hot-Dip) on Iron or Steel Hardware were prepared by the Section on Lead-Coated Hardware under the chairmanship of B. J. Barmack in order to conserve other metallic-coating materials. These specifications were based on considerable research and test work carried out by the Bell Telephone Laboratories, Western Electric Co., and the Commonwealth Edison Co. They were accepted by Committee E-10 on April 6, 1942, under the emergency procedure, and published under the designation ES-2.[1]

RECOMMENDATIONS AFFECTING STANDARDS

It has been decided that no changes are needed in the standards sponsored by this committee and it is therefore recommended that these standards be reprinted in the 1942 Book of A.S.T.M. Standards in their present form. The results of the letter ballot vote in the committee on continuing the specifications and recommended practice in their present status without revision are given in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Item	Affirmative	Negative	Ballots Marked "Not Voting"
I. REAFFIRMATION OF STANDARDS			
Spec. for Zinc-Coated (Galvanized) Iron or Steel Sheets (A 93 – 27)	68	0	13
Spec. for Zinc-Coated (Galvanized) Iron or Steel Telephone and Telegraph Line Wire (A 111 – 33)	64	0	17
Spec. for Zinc-Coated (Galvanized) Iron or Steel Tie Wire (A 112 – 33)	62	0	19
Spec. for Zinc-Coated Iron or Steel Chain-Link Fence Fabric Galvanized After Weaving (A 117 – 33)	60	0	21
Spec. for Zinc (Hot-Galvanized) Coatings on Structural Steel Shapes, Plates, and Bars, and Their Products (A 123 – 33)	65	0	16
Rec. Practice for Safeguarding Against Embrittlement of Hot-Galvanized Structural Steel Products and Procedure for Detecting Embrittlement (A 143 – 35)	68	0	13
II. TENTATIVE STANDARDS CONTINUED AS TENTATIVE WITHOUT CHANGE			
Spec. for Zinc-Coated (Galvanized) Iron or Steel Sheets (A 93 – 38 T)	68	0	13
Spec. for Electrodeposited Coatings of Zinc on Steel (A 164 – 40 T)	67	0	14
Spec. for Electrodeposited Coatings of Cadmium on Steel (A 165 – 40 T)	65	0	16

ACTIVITIES OF SUBCOMMITTEES

Subcommittee III on Inspection of Annapolis Tests (E. S. Taylerson, chairman).—The specimens of copper-bearing and non-copper-bearing corrugated black sheets that have been exposed at Annapolis, Md., since October 17, 1916, were inspected on May 6 and October 10,

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[1] These emergency specifications appear in the 1942 Book of A.S.T.M. Standards, Part I, p. 950.

TABLE II.—RECORD OF FAILURES IN ATMOSPHERIC EXPOSURE TESTS AT ANNAPOLIS OF NO. 22-GAGE COPPER-BEARING AND NON-COPPER-BEARING CORRUGATED BLACK SHEETS.

Sheets Exposed October 17, 1916. Inspections Made Semiannually from 1917 to 1942.

Group	Designation	Series	Average Analysis, per cent						Number of Sheets in Test[a]	Life of Each Failed Sheet, yr.																Number of Sheets in Test[a]	Number of Sheets Failed	Average Life of Sheets, yr.	Group
			Carbon	Manganese	Phosphorus	Sulfur	Silicon	Copper																					
Z......	Open-hearth steel	B	0.032	0.32	0.063	0.042	..	0.019	3	13.5	17.0	19.0	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	16.5	......Z
O......	Open-hearth steel	A	0.121	0.536	0.008	0.030	0.249	0.020	12	7.0	9.0	9.0	9.0	9.0	9.5	10.0	10.0	11.0	11.0	13.0	17.0	..	..	..	..	12	12	10.4	......O
B......	Open-hearth steel	A	0.09	0.38	0.017	0.062	0.036	0.084	3	20.0	20.5	24.0	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	21.5	......B
M......	Open-hearth steel	B	0.093	0.419	0.013	0.041	0.011	0.185	13	20.0	20.5	23.5	23.5	23.5	24.0	24.0	24.0	24.0	24.0	24.0	24.0	25.0	..	..	..	13	13	..	......M
B......	Open-hearth steel	A	0.06	0.27	0.014	0.053	0.006	0.187	9	19.5	20.0	21.5	22.0	22.5	23.0	*	*	*	..	..	..	..	..	..	..	9	6	..	......B
Z......	Open-hearth steel	B	0.067	0.351	0.045	0.052	0.003	0.187	3	*	*	*	..	..	..	..	..	..	..	..	..	..	..	..	..	3	0	..	......Z
T......	Open-hearth steel	B	0.083	0.313	0.009	0.020	0.005	0.227	14	19.5	19.5	19.5	21.5	21.5	21.5	23.0	23.5	23.5	23.5	24.0	24.0	24.0	24.0	..	..	14	14	22.3	......T
K......	Open-hearth steel (acid)	A	0.107	0.447	0.091	0.046	0.004	0.237	14	24.5	*	*	*	*	*	*	*	*	*	*	*	*	*	..	..	14	1	..	......K
H......	Open-hearth steel	A	0.069	0.387	0.016	0.027	0.004	0.244	10	19.5	21.0	21.5	23.0	23.5	23.5	23.5	24.0	24.0	25.0	..	..	..	..	..	..	10	10	..	......H
Z......	Open-hearth steel	B	0.065	0.35	0.050	0.059	0.003	0.264	2	24.5	*	..	..	..	..	..	..	..	..	..	..	..	..	..	..	2	1	..	......Z
Z......	Open-hearth steel	B	0.073	0.42	0.052	0.054	Trace	0.641	3	*	*	*	..	..	..	..	..	..	..	..	..	..	..	..	..	3	0	..	......Z
S......	Open-hearth iron	B	0.02	0.023	0.006	0.022	0.004	0.022	14	7.0	8.0	8.5	9.0	9.5	10.0	11.0	11.5	11.5	12.5	12.5	12.5	12.5	12.5	..	..	14	14	10.6	......S
D......	Open-hearth iron	A	0.02	0.026	0.004	0.022	0.002	0.024	6	16.0	16.0	16.0	19.5	19.5	23.5	..	..	..	..	..	..	..	..	..	..	6	6	18.4	......D
X......	Open-hearth iron	B	0.021	0.055	0.007	0.031	0.004	0.027	14	10.5	13.0	13.5	14.5	14.5	16.0	16.0	16.5	16.5	17.0	17.5	17.5	18.0	19.0	..	..	14	14	15.7	X
C......	Open-hearth iron	A	0.01	0.028	0.002	0.027	0.002	0.141	3	17.5	20.5	23.5	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	20.5	C
C......	Open-hearth iron	A	0.015	0.028	0.006	0.036	0.003	0.194	9	16.5	16.5	18.5	19.0	19.0	20.0	20.5	21.5	21.5	..	..	..	..	..	..	..	9	9	19.2	C
U......	Open-hearth iron	B	0.017	0.074	0.008	0.037	0.005	0.260	13	15.5	16.5	19.0	19.0	19.0	19.0	19.0	19.5	20.0	20.0	21.0	21.0	22.5	..	..	..	13	13	19.3	......U
V......	Open-hearth iron	B	0.018	0.051	0.007	0.021	0.005	0.304	14	17.0	17.0	17.5	18.5	18.5	19.0	20.0	20.0	20.5	21.0	22.0	22.0	22.5	24.0	..	..	14	14	20.0	V
A......	Bessemer steel	A	0.038	0.386	0.089	0.040	0.007	0.014	15	15.5	16.5	16.5	17.0	17.0	17.0	18.0	19.0	19.0	19.5	19.5	19.5	19.5	19.5	20.0	..	15	15	18.2	......A
Z......	Bessemer steel	B	0.063	0.41	0.102	0.044	0.009	0.013	3	11.5	11.5	11.5	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	11.5	......Z
Z......	Bessemer steel	B	0.077	0.44	0.095	0.036	0.003	0.140	3	*	*	*	..	..	..	..	..	..	..	..	..	..	..	..	..	3	0	..	......Z
I......	Bessemer steel	A	0.041	0.365	0.097	0.068	0.008	0.252	15	24.0	*	*	*	*	*	*	*	*	*	*	*	*	*	*	..	15	1	..	I
Z......	Bessemer steel	B	0.058	0.37	0.093	0.043	0.009	0.269	3	*	*	*	..	..	..	..	..	..	..	..	..	..	..	..	..	3	0	..	......Z
Z......	Bessemer steel	B	0.055	0.38	0.110	0.046	..	0.527	3	*	*	*	..	..	..	..	..	..	..	..	..	..	..	..	..	3	0	..	......Z
Y......	Wrought iron	B	0.03	0.055	0.139	0.021	0.218	0.020	3	18.0	19.5	23.5	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	20.3	......Y
E......	Wrought iron	A	0.033	0.034	0.114	0.021	0.134	0.283	10	19.5	20.5	21.0	21.0	23.0	23.0	23.0	23.0	24.0	25.0	..	..	..	..	..	..	10	10	..	......E
								Total	214																Total	214	168		

* Indicates unfailed sheet.

[a] The number of sheets in the test indicated in this table is less than the number shown in earlier reports because certain sheets were discarded due to mechanical damage.

1941. At the May inspection two No. 22 gage sheets had failed (K11, Z603) in addition to three No. 16 gage low-copper bessemer sheets (AA2, AA5, AA7). These are the first failures of No. 16 gage sheets. In October three No. 22 gage sheets had failed (E5, H14, M6). A summary of the failures to date is given in the accompanying Table II.

Subcommittee V on Total Immersion Tests (W. J. Jeffries, chairman).—It has been found impossible to complete the final report, as was promised in the annual report of the committee for 1941, summarizing the results obtained from all of the tests carried out by the subcommittee.

Subcommittee VI on Specifications for Metallic-Coated Products (T. R. Galloway, chairman).—As indicated earlier in this report, the subcommittee has reviewed standards under its jurisdiction and has recommended that they be reprinted in the new Book of Standards in their present form. Present-day conditions have necessarily slowed up the work of the subcommittee so that work which the subcommittee had hoped to complete for this meeting has not yet been finished. The status of the specifications for chain link fence (A 117 - 33) and for galvanized sheets (A 93 - 38 T) remain essentially the same as was reported in the last annual report. At the meeting in January, the Section on Sheets was authorized to proceed with the preparation of specifications for terne-coated sheets.

The Section on Lead-Coated Hardware (B. J. Barmack, chairman) prepared the Emergency Specifications for Lead Coating (Hot-Dip) on Iron or Steel Hardware which has been published by the Society under the designation ES-2.[1]

The Section on Galvanized Hardware (B. J. Barmack, chairman) is actively engaged in the revision of the Tentative Specifications for Zinc-Coating (Hot-Dip) on Hardware and Fastenings (A 153 - 33 T) and hopes to have the revised specifications ready for action later in the year.[2]

Subcommittee VII on Method of Testing (R. W. Baker, chairman).—Comments have been received on the Standard Method of Test for Uniformity of Coating by the Preece Test (Copper Sulfate Dip) on Zinc-Coated (Galvanized) Iron or Steel Articles (A 239 - 41), indicating that the description of the method for determining the end point for tests on wire is ambiguous. The committee has, therefore, referred this matter to the Preece Test Subgroup for action.

A section of the subcommittee headed by W. E. Buck was appointed to investigate the demand for and relative merits of methods for making the dropping test for determining the thickness of zinc and cadmium coatings on steel. Although this type of test was developed originally for use in connection with electrodeposited coatings, the section investigated its use also on hot-dip zinc coatings. Some consideration was given to the magnetic test method for determining thickness of protective metallic coatings. The report which was made at the January meeting of the subcommittee was considered of such general interest that it was published in the ASTM BULLETIN[3] as information in order to bring out suggestions for further development.

Subcommittee VII has reviewed the status of the standards under its jurisdiction and finds no changes necessary and recommends that they be published in their present form.

Subcommittee VIII on Field Tests of Metallic Coatings (R. F. Passano, chairman).—This subcommittee has continued its inspection of sheets, hardware, and wire. It had been planned to bring the tabulations showing data on the rusting of coated hardware, structural shapes, tubular goods, etc., up to date in this report. Considering the demands that have been made on the members by things more important to the war effort, the subcommittee has postponed the extension of these tabulations until another time.

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[2] See Editorial Note, p. 119.

[3] W. E. Buck, "Report on Dropping Tests for Zinc and Cadmium Coatings," ASTM BULLETIN, No. 115, March, 1942, p. 33.

The report on the exposure tests of galvanized sheets is appended hereto.

The following officers of Committee A-5 have been elected for the ensuing term of two years:

Chairman, C. D. Hocker.
First Vice-Chairman, R. F. Passano.
Second Vice-Chairman, T. R. Galloway.

This report has been submitted to letter ballot of the committee, which consists of 130 members, 81 members returned their ballots, of whom 78 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. H. FINKELDEY,
Chairman.

T. R. GALLOWAY,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee A-5 presented to the Society through Committee E-10 on Standards a revision of the Tentative Specifications for Zinc Coating (Hot-Dip) on Hardware and Fastenings (A 153 – 33 T). The revised tentative specifications were accepted by Committee E-10 on August 24, 1942, and appear in the 1942 Book of A.S.T.M. Standards, Part I, p. 1066.

REPORT OF SUBCOMMITTEE VIII ON FIELD TESTS OF METALLIC COATINGS

TESTS OF CORRUGATED GALVANIZED SHEETS

Galvanized Coatings on Corrugated Sheets at Sandy Hook:

At Sandy Hook, N. J., one galvanized sheet specimen in the No. 22 gage, 2.50 oz. per sq. ft. class, developed rust after 14.40 yr. This is the only specimen that has rusted at this location since the 1940 report.[1] There remain three unrusted specimens in this coating class and these are the only unrusted ones remaining at this location. Inasmuch as the change is minor, no tabulation for this location is shown in this report. For a detailed account of previous coating failures reference should be made to the 1940 report[1] and the earlier tabulations referred to therein.

The rack at Sandy Hook has been inspected twice each year since the galvanized sheet test was started in 1926. One might imagine that approximately one half of the specimens would have been discovered with rust at the inspections made in April and the other half at the inspections made in October. Actually 81 of the 105 specimens now showing rust have been discovered at the October inspections; this 77 per cent of 105 specimens is scarcely a chance variation of the 50 per cent expected. It was postulated that the atmosphere at Sandy Hook corroded zinc more rapidly between April and October than it did between October and April.

Loss of Weight Experiment at Sandy Hook:

A loss of weight experiment was accordingly started in April, 1939. Briefly, 4 by 6-in. specimens of galvanized sheets and 4 by 6-in. specimens of rolled zinc sheet were exposed vertically and at 30 deg. from the horizontal. Vertical exposure has been used at Sandy Hook by Subcommittee VI on Atmospheric Corrosion of Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys and in the tests being carried on in cooperation with the British Iron and Steel Institute. Exposure at 30 deg. from the horizontal has been standard in all atmospheric corrosion tests on sheets of Committee A-5. The composition of the test specimens involved in these loss of weight determinations is shown in Table I.

TABLE I.—CHEMICAL COMPOSITION OF ZINC IN LOSS OF WEIGHT EXPERIMENT AT SANDY HOOK, N. J.

	Rolled Zinc Sheet	Zinc Coating on Galvanized Sheet[a]
Iron, per cent	0.025	2.84[b]
Tin, per cent	0.005	0.13
Lead, per cent	0.74	0.81
Cadmium, per cent	<0.001	0.32

[a] 2.25 oz. per sq. ft. of sheet, or, 10.61 g. per 4 by 6-in. specimen.

[b] 0.064 oz. per sq. ft. of sheet.

All specimens were cleaned and weighed before exposure. After exposure, they were cleaned free of corrosion products and reweighed. So far as is known, the cleaning loss is of the order of 1 mg. per specimen and is included in the loss of weight data presented in Table II. The data in Table II have been condensed in accordance with the recommendations of Technical Committee IX on Interpretation and Presentation of Data, of Committee E-1 on Methods of Testing.[2]

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 106 (1940).

Only a small fraction of the zinc available on the specimen has been used in these tests. It appeared that the specimens exposed vertically had lost weight about equally from the two surfaces. The specimens exposed at 30 deg. corroded almost entirely on the upper or unsheltered surface. The sheared edges of the galvanized sheet specimens after exposure were found covered for the most part with what appeared to be corrosion products of zinc. These were tinged yellow with iron salts in some places. There was no accumulation of typical base metal rust on the edges at any place. There was no evidence of unusual attack on the galvanized surfaces adjacent to the sheared edges. There were, however, relatively heavy rings of zinc corrosion products around the points where the test specimens of both rolled zinc and galvanized sheet were supported. Six grooved, cylindrical porcelain insulators about $\frac{3}{4}$ in. in diameter were in possible contact with each 4 by 6-in. specimen.

TABLE II.—LOSS OF WEIGHT IN MILLIGRAMS FROM 4 BY 6-IN SPECIMENS EXPOSED AT SANDY HOOK, N. J.

Exposed	Removed	Length of Exposure	Material	Exposed at 30 deg. from Horizontal			Exposed Vertically		
				Number of Specimens	Arithmetic Mean, $\overline{X}$	Standard Deviation, σ	Number of Specimens	Arithmetic Mean, $\overline{X}$	Standard Deviation, σ
April 18, 1939	Oct. 11, 1939	176 days	Galvanized steel sheet..	5	61.4	5.1	5	31.4	6.8
April 18, 1939	Oct. 11, 1939	176 days	Zinc sheet............	5	60.6	4.7	5	32.4	4.0
Oct. 11, 1939	April 30, 1940	202 days	Galvanized steel sheet..	5	53.8	4.0	5	32.4	5.9
Oct. 11, 1939	April 30, 1940	202 days	Zinc sheet............	[a]	[a]	[a]	5	31.0	4.0
April 18, 1939	April 30, 1940	378 days	Galvanized steel sheet..	5	136.4	4.6	5	69.8	5.0
April 18, 1939	April 30, 1940	378 days	Zinc sheet............	2	135.5	6.5	5	68.2	7.9
Oct. 11, 1939	Oct. 10, 1940	365 days	Galvanized steel sheet..	4	485.5	14.5	5	426.6	7.5
Oct. 11, 1939	Oct. 10, 1940	365 days	Zinc sheet............	3	408.7	3.1	5	379.0	8.4
April 30, 1940	Oct. 10, 1940	163 days	Galvanized steel sheet..	4	347.2	8.6	4	312.2	12.5
April 30, 1940	Oct. 10, 1940	163 days	Zinc sheet............	5	279.8	11.4	2	253.5	12.5

[a] This sample lost from racks.

It seems that more questions have been raised by this experiment than have been answered by it. The losses shown for the April to October, 1940, period and for the October, 1939, to October, 1940, period are very different from losses for the earlier periods. This, it is believed, is due to temporary circumstances which did not exist between 1926 and 1940.

In regard to the speculations which started the loss of weight experiment, there remain few data to settle the matter. The annual rate of loss for the period April to October, 1939, was 126.5 mg. per specimen for the samples exposed at 30 deg. and 66.1 mg. per specimen for those exposed vertically. Between October, 1939, and April, 1940, the annual rate of loss for the sample exposed at 30 deg. was 97.1 mg. per specimen and 57.3 mg. per specimen for those exposed vertically.

Considering these two periods only,

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[2] For method of presentation, see A.S.T.M. Manual on Presentation of Data, *Proceedings*, Vol. 33, Part I, p. 451 (1933). (Also available as separate publication.)

the October to April rate is 81.6 per cent (average of 76.6 and 86.6 per cent) of the April to October rate. The data produced by this loss of weight experiment are in line with the speculations based on the observations on sheets.

TABLE III.—RECORD OF COATING FAILURES ON STATE COLLEGE, PA., TEST RACK.
SHEETS EXPOSED APRIL 23, 1926.

Description	Number of Specimens in Test	Up to Oct.6, 1939,[a] 13.49 yr.	April, 1940, 14.03 yr.	Oct., 1940, 14.47 yr.	April, 1941, 15.03 yr.	Oct., 1941, 15.46 yr.	Total Number of Failures	Average Life of Coating in Yr.
No. 16 gage, 2.50 oz. per sq. ft.	12	none	none	none	none	none	none	....
No. 22 gage, 2.50 oz. per sq. ft.	18	none	none	none	none	none	none	....
No. 22 gage, 2.00 oz. per sq. ft.	18	none	none	none	none	none	none	....
No. 22 gage, 1.50 oz. per sq. ft.	18	none	1	none	1	2	4	....
No. 22 gage, 1.25 oz. per sq. ft.	18	4	none	7	4	1	16	(14.47)[b]
No. 22 gage, 0.75 oz. per sq. ft.	14	14	....	....	....	....	14	10.04
No. 28 gage, 0.75 oz. per sq. ft.	10	10	....	....	....	....	10	11.20
Total	108	28	1	7	5	3	44	....

[a] A detailed record of the coating failures observed at the inspections of April, 1938, to October, 1939, inclusive, appears in Table III of the 1940 Report, see *Proceedings*, Vol. 40, p. 107 (1940). For a record of coating failures observed up to October, 1937, see *Proceedings*, Vol. 38, Part I, p. 87 (1938).

[b] Figures shown in parentheses are not to be considered final.

TABLE IV.—TIME IN YEARS AT WHICH THERE WAS FIRST INDICATION OF PERFORATION OF GALVANIZED SHEETS ON KEY WEST, FLA., TEST RACK.
SHEETS EXPOSED JUNE 9, 1926.

Description	Number of Specimens in Test	Jan., 1937, 10.64 yr.	March, 1938, 11.79 yr.	March, 1939, 12.79 yr.	April, 1940, 13.84 yr.	April, 1941, 14.81 yr.	Total Number of Failures	Average Life of Sheet in Years
No. 16 gage, 2.50 oz. per sq. ft.	12	none	none	none	none	none	none	....
No. 22 gage, 2.50 oz. per sq. ft.	18	none	none	none	none	none	none	....
No. 22 gage, 2.00 oz. per sq. ft.	18	none	1	none	none	1	2	....
No. 22 gage, 1.50 oz. per sq. ft.	18	none	3	5	3	none	11	(14.7)[a]
No. 22 gage, 1.25 oz. per sq. ft.	18	2	4	4	2	2	14	(13.6)[a]
No. 22 gage, 0.75 oz. per sq. ft.	14	5	4	8	none	none	12	(12.1)[a]
No. 28 gage, 0.75 oz. per sq. ft.	10	2	3	2	1	1	9	(12.7)[a]
Total	108	9	15	14	6	4	48	

[a] Figures shown in parentheses are not to be considered final.

Those specimens that were exposed for periods of a year, from April to April and from October to October were paralleled, as it were, by specimens exposed for 6 months, from April to October, 1939, from October, 1939, to April, 1940, and April to October, 1940. In every case the sum of losses for the two proper half-year periods is less than the loss for a one-year period. The sum of losses for two half-year periods averages 84.2 per cent of the loss for the one year period. This can be explained in several ways any of which would be speculative.

In eight of the nine cases where losses on rolled zinc and galvanized coating are available, the rolled zinc has lost less weight than the galvanized coating. The difference is negligible in five of the nine cases.

Galvanized Coatings on Corrugated Sheets at State College:

Sixteen corrugated specimens at State College, Pa., have developed rust since the 1940 report. These data are shown in Table III.

Galvanized Coatings on Corrugated Sheets at Key West:

It will be recalled that deterioration of the sheets at Key West, Fla,, takes place from the under surface of a sheet while the upper, exposed, and unsheltered surface remains thoroughly covered by zinc and its corrosion products. This is in contrast to the developments at the four northern test locations where the first deterioration is that of the galvanized coating on the exposed surface. This condition still continues. The data now available from Key West, Fla., are shown in Table IV.

TESTS OF METALLIC-COATED HARDWARE

It had been planned to bring the tabulations showing data on the rusting of metallic-coated hardware, structural shapes, tubular goods, etc., up to date in this report. Considering the demands that have been made on the members by things more important to the war effort, the subcommittee has postponed the extension of these tabulations until another time.

Respectfully submitted on behalf of the subcommittee,

R. F. PASSANO,
Chairman.

REPORT OF COMMITTEE A-6
ON
MAGNETIC PROPERTIES

No meeting of Committee A-6 on Magnetic Properties has been held since the annual meeting of the Society in 1941, but Subcommittees III and IV have met and much work has been carried on by correspondence and conferences between small groups.

Mr. R. F. Field of the General Radio Co. has been appointed to membership on the committee. He has also been appointed a member of Subcommittee IV. Henry Wysor of the Bethlehem Steel Corp. has resigned.

The present officers of the committee and members of the Advisory Committee have been re-elected for the ensuing term of two years.

A number of investigators have attempted, over a period of a good many years, to develop a satisfactory method of measuring the magnetic properties of single sheets and wide strips of electrical sheet. So far no satisfactory method has become available for making such tests.

During the past five or six years W. E. Shenk and his associates at the Research Laboratory of the United States Steel Corp. have been developing a novel method for the magnetic testing of full-sized sheets of various widths. A number of these devices have recently been installed in commercial steel mills. A paper by W. E. Shenk on "The Absolute Measurement of the Energy Loss in a Single Strip of Sheet Iron or Steel Under Alternating Magnetization" describing this new magnetic testing device appears in the Appendix[1] to this report. Some day this or similar methods may partly or wholly supplant the commercial methods of sampling and testing, using Epstein or other equipment employing narrow samples. Two major advantages of such a test are: (*a*) The elimination of punching or shearing strain effects, and (*b*) a reduction in the cost of sampling. It seems very desirable, therefore, that the A.S.T.M. consider and publicize this new development.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption the following revisions in the Standard Methods of Test for Magnetic Properties of Iron and Steel (A 34 – 41),[2] and accordingly asks for a favorable nine-tenths vote at the annual meeting in order that these modifications may be referred to letter ballot of the Society.

Section 3 (*d*).—Add the following as a second paragraph to the Note at the end of this section:

> Where tests cannot be made by using Epstein specimens, in cases where it is desired to determine the degree of directional magnetic properties, a ring specimen may be used and compared with tests made on an Epstein specimen the length of which is parallel to the rolling direction.

Section 9.—Change the title of this section to read as follows by the addition of the italicized words and figures:

Apparatus (*50 cm.* Epstein, *Butt Joint*)

Section 18.—Change the title of this section to read as follows by the addition of the italicized words and figures:

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[1] See p. 595.

[2] 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 121.

Apparatus, Bridge Method (*25-cm. Epstein, Double Lap Joint*)

Section 19 (c).—In order to correct a minor error in various equations for calculating μ, substitute "0.748" for "0.747" and "7.48" for "7.47".

Add the following equation after the equation for core loss, P:

and the Vars:

$$\text{Vars} = \frac{E^2 \omega C_3 R_1}{R_4}$$

After the definition for R_4, add the following definition for Vars:

Vars = total reactive volt-amperes.

Change Note 1 to read as follows:

NOTE 1.—P and Vars, divided by 0.84 times the mass in kilograms gives the loss in watts per pound and the reactive volt-amperes in Vars per pound, respectively.

Section 20.—Change the title of this section to read as follows by the addition of the italicized words and figures:

Apparatus, Potentiometer Method (*25-cm. Epstein, Double Lap Joint*)

Section 21 (d).—After the equation for P, add the following equation:

and:

$$\text{Vars} = \frac{N_1}{N_2} E I_Q$$

The term Vars is introduced because many designers prefer to use this unit rather than permeability.

With the inclusion of various lengths of Epstein specimens, in addition to the original 50-cm. length, it seems desirable to designate more accurately the length of specimens and the type of joint for the various test methods. For these reasons the changes in the titles of Sections 9, 18, and 20 are recommended.

The proposed addition to the Note in Section 3 (*d*) is for the purpose of providing a means of determining the degree of directional magnetic properties in sheet or strip which are so narrow that crosswise 25-cm. length specimens cannot be cut from the strip. A large amount of this narrow strip is now being produced.

This recommendation has been submitted to letter ballot of the committee which consists of 24 members; 18 members returned their ballots of whom 18 have voted affirmatively, 0 negatively, and 0 members marked their ballots "not voting."

ACTIVITIES OF SUBCOMMITTEES

Subcommittee III on Direct Current Test Methods (B. M. Smith, chairman).—This subcommittee has been working for about two years on the development of Methods for Test for Permeability of Feebly Magnetic Materials (the so-called nonmagnetic steels) having permeabilities not exceeding 2. These methods include two procedures, the second of which covers the Fahy Low-Mu Permeameter which has certain patented features. It is expected that a third test procedure, which was brought to the attention of the committee so recently that it could not be completed for inclusion at this time, will subsequently be added to the methods. It was expected that these methods would be offered to the Society at the annual meeting for publication as tentative. However, in attempting to obtain written approval by the committee so many new suggestions were received that the methods are being published as information as appended hereto,[3] with the expectation that they will be presented to the Society subsequent to the June meeting through Committee E-10 on Standards for acceptance as tentative.[3]

Subcommittee IV on Alternating Current Test Methods (J. P. Barton, chairman).—As mentioned in the report last year, this subcommittee has been working for some time on two new methods of test, namely, a Proposed Method of

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[3] These methods were accepted as tentative by Committee E-10 on Standards, see Editorial Note, p. 126.

Test for Permeability and Core Loss of Flat-Rolled Magnetic Materials Using 28-cm. Specimen, and a Proposed Method of Test for Incremental Permeability and Core Loss of Flat-Rolled Magnetic Materials at Low Alternating Inductions Using 28-cm. Specimen.

This work has progressed to such a point that it is now possible to offer these proposed methods to the Society for information and discussion as appended hereto.[3] If there are not too many suggested changes, these test methods will be submitted to Committee E-10 for acceptance as tentative standards later this year.[3] Due to the pressure of war work, the number of check tests on these methods, as originally scheduled, have not been completed and may have to be delayed for the duration. However, sufficiently good agreement among the various laboratories which have participated indicates that the methods are fundamentally sound.

One of the chief expected advantages of the first method is that it probably will be possible to make alternating current and direct current check tests on the same sample in the same test frame. Further work is contemplated to determine the degree of agreement between alternating current and direct current tests, using this method.

The second method is designed primarily for testing material to be used in the cores of radio and communication transformers and chokes. No standard method is now available in this country for determining these incremental magnetic properties.

It should be noted that this test equipment is based on the bridge method already adopted for making alternating current tests at low inductions and, so far as possible, use is made of the standard bridge arrangement. It may also be pointed out that by simple modification the potentiometer method of making low induction alternating current tests[4] can be used for determining the incremental magnetic properties. The committee has not yet experimented with such a method.

This report has been submitted to letter ballot of the committee which consists of 24 members; 18 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

THOMAS SPOONER,
Chairman.

R. L. SANFORD,
Secretary.

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[4] See Fig. 3 of the Standard Methods of Test for Magnetic Properties of Iron and Steel (A 34 - 41), 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 128.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee A-6 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative Methods of Test for:

Permeability and Core Loss of Flat-Rolled Magnetic Materials Using 28-cm. Specimen (A 257 - 42 T),

Incremental Permeability and Core Loss of Flat-Rolled Magnetic Materials at Low Alternating Inductions Using 28-cm. Specimen (A 258 - 42 T), and

Permeability of Feebly Magnetic Materials (A 259 – 42 T).

These new tentative methods were accepted by Committee E-10 on August 24, 1942, and assigned the A.S.T.M. serial designations indicated above. These tentative standards appear in the 1942 Book of A.S.T.M. Standards, Part I, pp. 1091, 1097, and 1103.

REPORT OF COMMITTEE A-7

ON

MALLEABLE-IRON CASTINGS

Committee A-7 on Malleable Iron Castings has held two meetings during the past year: one on June 26, 1941, during the annual meeting of the Society in Chicago, Ill., and the other in Cleveland, Ohio, on March 3, 1942, in conjunction with the spring group meetings.

Vice-Chairman J. J. Kanter has acted as chairman during Mr. Wolf's absence in Washington.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, E. K. Smith
Vice-Chairman, J. J. Kanter
Secretary, H. A. Schwartz

Subcommittee III on Cupola Malleable Iron (J. J. Kanter, chairman), following its meeting in March, presented to Committee A-7 proposed tentative specifications for malleable iron castings for flanges, pipe fittings, and valve parts and subsequently presented amendments of an editorial nature. These proposed specifications were submitted to letter ballot of the committee but, although they received the required two-thirds affirmative vote, the objections and discussions were of such a character that Subcommittee III asked that the proposed specifications be referred back for further consideration and amendment.[1]

Subcommittee V on Air Furnace and Electric Malleable Iron (J. H. Lansing, chairman) also met in March and reported progress but is not yet prepared to submit a proposed specification for air furnace and electric malleable iron castings.

On the recommendation of Committee A-7, the following standards were submitted to the American Standards Association for approval as American Standard:

A.S.T.M. Standard Specifications for:
Malleable Iron Castings (A 47 – 33), and
Cupola Malleable Iron (A 197 – 39).

These were approved by the ASA on June 5, 1942, and assigned the ASA designations G48.1-1942 and G49.1-1942, respectively.

This report has been submitted to letter ballot of the committee, which consists of 40 members, 36 members returned their ballots, of whom 27 have voted affirmatively, and 0 negatively.

Respectfully submitted on behalf of the committee,

F. L. Wolf,
Chairman.

H. A. Schwartz,
Secretary.

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[1] See Editorial Note, below.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee A-7 submitted to Committee E-10 on Standards proposed Emergency Specifications for Malleable Iron Flanges, Pipe Fittings, and Valve Parts. This recommendation was accepted by Committee E-10 on October 6, 1942, and the emergency standard appears in the 1942 Book of A.S.T.M. Standards, Part I, p. 959, bearing the A.S.T.M. serial designation ES-20.

REPORT OF COMMITTEE A-9
ON
FERRO-ALLOYS

Committee A-9 on Ferro-Alloys has held one meeting since its previous report to the Society in 1941: in Cleveland, Ohio, on March 3, 1942, in conjunction with the spring group meetings.

The committee discussed the possibility of the need for emergency standards or emergency alternate provisions in its existing specifications during the war. This matter was carefully considered and it was agreed that in view of the broad tolerances in the present specifications under the jurisdiction of Committee A-9 no action is required, and no possible necessary action is indicated within the next two years.

Subcommittee I on Specifications, under the chairmanship of E. A. Lucas, has by personal contact and correspondence continued an examination of the possibilities of preparing tentative specifications for ferroboron, ferrotitanium, ferrocolumbium, etc. In view of the lack of experience with these materials, it was the consensus that an attempt should not be made at this time to prepare specifications in this field.

ADOPTION OF TENTATIVE STANDARD AS STANDARD

Tentative Specifications for Ferrochromium (A 101 – 39 T).[1]—As reported last year, the results of the letter ballot vote on the adoption as standard of the Tentative Specifications for Ferrochromium were as follows: From a membership of 27, all the members returned their ballots, 26 of whom voted affirmatively and one negatively. This negative vote has since been withdrawn and it is therefore recommended that these tentative specifications be approved for reference to letter ballot of the Society for adoption as standard.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, J. H. Critchett
Vice-Chairman, C. M. Loeb, Jr.
Secretary, A. P. Spooner

This report has been submitted to letter ballot of the committee which consists of 27 members; 23 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

CHARLES MCKNIGHT,
Chairman.

C. M. LOEB, JR.,
Secretary.

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[1] 1939 Book of A.S.T.M. Standards, Part I, p. 986.

REPORT OF COMMITTEE A-10

ON

IRON-CHROMIUM, IRON-CHROMIUM-NICKEL AND RELATED ALLOYS

Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys held two meetings during the year: in Chicago, Ill., on June 25, 1941, and in Cleveland, Ohio, on March 3, 1942.

There were several resignations and changes in committee personnel during the year. The present membership is 78 of whom 34 are classified as producers, 32 as consumers, and 12 as general interest members.

The present officers of Committee A-10 have been re-elected for the ensuing term of two years. Elections in the subcommittees resulted in the re-election of E. F. Cone, as chairman, and Russell Franks, as secretary, of Subcommittee I; F. L. LaQue was appointed chairman of Subcommittee IV succeeding E. S. Taylerson; L. C. Bibber was re-elected chairman of Subcommittee VII; J. J. Kanter was appointed chairman of Subcommittee X, succeeding W. J. Jeffries who resigned as chairman of this subcommittee; and J. K. Findley was appointed chairman of Subcommittee VIII, succeeding H. S. Schaufus who resigned the chairmanship of this subcommittee. A. B. Bagsar accepted membership on the Joint American Welding Society-A.S.T.M. Committee on Filler Metal, representing Committee A-10.

EMERGENCY ALTERNATE PROVISIONS

During the year, Committee A-10 submitted to Committee E-10 on Standards Emergency Alternate Provisions in the following specifications:

Standard Specifications for:

Corrosion-Resisting Chromium-Nickel Steel Sheet, Strip, and Plate (A 167 – 41), and

Corrosion-Resisting Chromium-Nickel Steel Sheet, Strip, and Plate for Fusion-Welded Unfired Pressure Vessels (A 240 – 41).

These Emergency Alternate Provisions were accepted by Committee E-10 on April 6, 1942. Upon further recommendation of Committee A-10, additional Emergency Alternate Provisions in the above standards as well as in the Standard Specifications for High-Strength Corrosion-Resisting Chromium-Nickel Steel Sheet and Strip (A 177 – 39) were accepted by Committee E-10 on May 7, 1942. The provisions approved in May provide for increasing the limits on maximum content of sulfur and phosphorus from 0.030 and 0.035 per cent to a value of 0.040 per cent because of melting and scrap limitations. These Emergency Provisions are likewise in line with action taken by the Stainless Steel Technical Committee of the American Iron and Steel Institute. The alternate provisions approved in April as well as those accepted in May have been published in the form of "pink stickers" for attachment to the specifications to which they apply, under the following designations: EA - A 167, EA - A 177, and EA - A 240.

REVISIONS OF STANDARDS

The committee recommends for immediate adoption, revisions in three specifications, as indicated below, and accordingly asks for the necessary nine-tenths vote at the annual meeting in order that these modifications may be referred to letter ballot of the Society:

Standard Specifications for Corrosion-Resisting Chromium-Nickel Steel Sheet, Strip, and Plate (A 167 – 41):[1]

Table I.—Make the following changes in the requirements as to chemical composition:

Grade	Type Number	Element	Change Present Requirement From, per cent	To Read, per cent
1	301	Manganese	1.50 max.	2.00 max.
		Chromium	16.00 min.	16.00 to 18.00
		Nickel	7.00 min.	6.00 to 8.00
2	302	Manganese	1.50 max.	2.00 max.
		Chromium	18.00 min.	17.00 to 19.00
		Nickel	8.00 min.	8.00 to 10.00
3	304	Manganese	1.50 max.	2.00 max.
		Chromium	18.00 min.	18.00 to 20.00
		Nickel	8.00 min.	8.00 to 10.00
4	308	Manganese	1.50 max.	2.00 max.
		Chromium	19.00 min.	19.00 to 22.00
		Nickel	10.00 min.	10.00 to 12.00
7	302 B	Manganese	1.50 max.	2.00 max.
		Chromium	18.00 min.	17.00 to 19.00
		Nickel	8.00 min.	8.00 to 10.00
8	309	Carbon	0.08 to 0.20	0.20 max.
		Chromium	22.00 min.	22.00 to 24.00
		Nickel	12.00 min.	12.00 to 15.00
9	309 S	Chromium	22.00 min.	22.00 to 24.00
		Nickel	12.00 min.	12.00 to 15.00
10	310	Chromium	24.00 min.	24.00 to 26.00
		Nickel	19.00 min.	19.00 to 22.00

Standard Specifications for Corrosion-Resisting Chromium Steel Sheet, Strip, and Plate (A 176 – 41):[2]

Table I.—Omit from this table all the requirements for grades 3 and 7, and make the following changes in the requirements as to chemical composition for grades 1, 2, and 4:

Grade	Type Number	Element	Change Present Requirement From, per cent	To read, per cent
1	403	Carbon	0.12 max.	0.15 max.
2	410	Carbon	0.12 max.	0.15 max.
		Chromium	12.00 to 14.00	10.00 to 14.00
4	430	Chromium	16.00 to 18.00	14.00 to 18.00

Table II.—Omit from this table all the tensile requirements for grades 3 and 7.

Standard Specifications for Corrosion-Resisting Chromium-Nickel Steel Sheet, Strip, and Plate for Fusion-Welded Unfired Pressure Vessels (A 240 – 41):[3]

Title.—Add the words "Chromium and" before the words "Chromium-Nickel Steel."

Table I.—Add the following requirements as to chemical composition for two new grades A and B:

	Grade A (Type 430)	Grade B (Type 446)
Carbon, max., per cent	0.12	0.35
Manganese, max., per cent	1.00	1.00
Phosphorus and sulfur, max., per cent	0.030	0.030
Silicon, max., per cent	1.00	1.00
Chromium, per cent	14.00 to 18.00	23.00 to 30.00
Nickel, max., per cent	0.60	0.60

Table II.—Add the following requirements as to tensile properties for two new grades A and B:

	Grade A (Type 430)	Grade B (Type 446)
Tensile strength, min., psi	70 000	75 000
Yield strength, min., psi	35 000	40 000
Elongation in 2 in. min., per cent	22.0[b]	20.0
Brinell number, max.	202	217
Rockwell number, max.	B 88	B 95
Cold bend, deg.	180	135

[b] Material 0.050 in. and under in thickness (U. S. gage No. 18 and under) shall have a minimum elongation of 20.0 per cent.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 78 members; 54 members returned their ballots, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
REVISIONS OF STANDARDS, IMMEDIATE ADOPTION			
Spec. for Corrosion-Resisting Chromium - Nickel Steel Sheet, Strip, and Plate (A 167 – 41)	43	0	11
Spec. for Corrosion-Resisting Chromium Steel Sheet, Strip, and Plate (A 176 – 41)	42	0	12
Spec. for Corrosion-Resisting Chromium - Nickel Steel Sheet, Strip, and Plate for Fusion - Welded Unfired Pressure Vessels (A 240 – 41)	42	0	12

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[1] 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 50.

[2] *Ibid.*, p. 57.

[3] *Ibid.*, p. 64.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Classification of Data (E. F. Cone, chairman; Russell Franks, secretary).—The data collected by this subcommittee have been compiled by Russell Franks and F. L. LaQue and are now being published by the Society in the form of "Tables of Data on Chemical Compositions, Physical and Mechanical Properties of Wrought Corrosion-Resisting and Heat-Resisting Chromium and Chromium-Nickel Steels."[4] It is divided into two parts, Part I covering the Wrought Chromium Steels and Part II Wrought Chromium-Nickel Steels.

Forms for collecting data on the alloys in cast form are being supplied by the Alloy Casting Institute. This information will be correlated and published at a later date.

Subcommittee IV on Methods of Corrosion Testing (F. L. LaQue, chairman). —This subcommittee has under consideration the preparation of a standard boiling nitric acid test. The formulation of a standard test procedure has been requested by the Welding Research Committee of the American Welding Society for use in connection with the testing of welds. A draft of a procedure for a general boiling liquid test may be modified to provide a procedure for the boiling nitric acid test.

Subcommittee V on Mechanical Tests.—(T. F. Olt, chairman).—Subcommittee V is securing information on the effect of rate of straining on the mechanical properties of the austenitic chromium-nickel-iron alloys. It will also study the variations that might be encountered in utilizing the methods of determining yield strength as described in the Tentative Methods of Tension Testing of Metallic Materials (E 8 - 40 T).[5] A round-robin series of tests is being organized among members of the subcommittee to obtain further data on the subject. The Section on Tension Testing, of Technical Committee I, of Committee E-1 on Methods of Testing, is being consulted in connection with the program which will be referred to Committee A-10 when it has been fully decided upon.

Subcommittee VI on Metallography (Russell Franks, chairman).—This subcommittee has completed its second report on "Metallographic Examination of 18 per cent Chromium 8 per cent Nickel Steel" which is appended to this report. In the light of discussions at a meeting held during this annual meeting the text will be revised and submitted to letter ballot in the committee for approval for publication.[6]

Subcommittee VII on Welding (L. C. Bibber, chairman).—The function of Subcommittee VII is primarily one of liaison between Committee A-10 and other research and specification-writing committees of the Engineering Foundation, the American Welding Society, and other agencies working on similar problems. This liaison has been carried out through attendance at most of the meetings of the committees involved. Because of the present intangible function of this subcommittee, consideration is being given to discharging it and handling the liaison work of Committee A-10 in the field of welding of high alloy steels by appointed representatives.

Subcommittee VIII on Specifications for Wrought Products.—This subcommittee is in process of reorganization and a new chairman, J. K. Findley, has recently been appointed. Consideration is still being given to the formulation of specifications covering hot-rolled and cold-drawn bars and also spring wire of the corrosion-resisting steels.

Subcommittee IX on Specifications for Flat Products (C. C. Snyder, chairman). —Subcommittee IX recommended

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[4] Issued December, 1942.

[5] 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 453.

[6] Of a total membership of 78, 54 members returned their ballots of whom 52 voted affirmatively, 1 negatively, and 1 member marked his ballots "not voting."

Emergency Alternate Provisions in the Specifications for Corrosion-Resisting Chromium-Nickel Steel Sheet, Strip, and Plate (A 167 – 41), for High-Strength Corrosion-Resisting Chromium-Nickel Steel Sheet and Strip (A 177 – 39), and for Corrosion-Resisting Chromium-Nickel Steel Sheet, Strip, and Plate for Fusion-Welded Unfired Pressure Vessels (A 240 – 41) which were accepted by Committee E-10 as noted earlier in this report. This action brings the specifications in line with existing Federal or Emergency Specifications and with the recommendations of the American Iron and Steel Institute.

The subcommittee is now recommending for immediate adoption changes in the requirements for chromium, nickel, and carbon in the Standard Specifications A 167, A 176, and A 240 in the interest of conserving critical metals. These revisions are covered earlier in this report. In the case of Specifications A 240, the two new grades A and B are being added at the request of the A.S.M.E. Boiler Code Committee.

A subgroup to draft specifications for stainless clad metals is being organized under the chairmanship of D. B. Rossheim. Specifications for this material were requested by the A. S. M. E. Boiler Code Committee as composite metals clad with stainless alloys are being used in equipment coming under code ruling.

Subcommittee X on Specifications for Castings (J. J. Kanter, chairman).—Further revisions of the specifications for castings prepared by this subcommittee are being held in abeyance pending collection of data on cast alloys through the Alloy Casting Institute. A joint meeting of the subcommittee and Committee B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys may be held in connection with questions arising on specifications for heat-resisting castings for furnace use. In such event, the specifications issued by Committee A-10 on cast alloys will need to be reviewed. The standards involved are the Specifications for Chromium Alloy-Steel Castings (A 221 – 39),[6] for 20 per cent Chromium, 9 per cent Nickel Alloy-Steel Castings (A 198 – 39),[6] and for Chromium-Nickel Alloy-Steel Castings (A 222 – 39).[7]

Subcommittee XI on Specifications for Tubular Products (Newell Hamilton, chairman).—This subcommittee has been relatively inactive during the past year. Several of its members have assisted in preparing specifications for tubular products in which the stainless steels are incorporated. These Specifications for Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F. (A 158 – 41 T) and for Seamless Alloy-Steel Boiler and Superheater Tubes (A 213 – 40) are under the jurisdiction of Committee A-1 on Steel. Other specifications covering tubular products of stainless steel are under consideration.

This report has been submitted to letter ballot of the committee which consists of 78 members; 54 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

JEROME STRAUSS,
Chairman.

H. D. NEWELL,
Secretary.

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[7] 1939 Book of A.S.T.M. Standards, Part I, pp. 381, 385, 390.

REPORT OF SUBCOMMITTEE VI ON METALLOGRAPHY

SECOND REPORT ON METALLOGRAPHIC EXAMINATION OF 18 PER CENT CHROMIUM, 8 PER CENT NICKEL STEEL

In the earlier Report on Metallographic Examination of 18 per cent Chromium, 8 per cent Nickel Steels[1] it was reported that two heats of austenitic chromium-nickel steels were studied metallographically by a number of cooperating laboratories, the purpose of the study being to determine the nature of the material precipitated at the grain boundaries when an austenitic stainless steel was subjected to heat treatment that renders the metal susceptible to intergranular attack. The original objective of this cooperative study was not obtained, however, in the first experiments; that is, practically no information was obtained on the nature of the precipitation that takes place in the grain boundaries of the 18 per cent chromium, 8 per cent nickel steel when it is heated at temperatures of 550, 650, 750, and 850 C., respectively.

In view of this, it was suggested to Subcommittee VI that heat No. 1 be examined after etching with sodium cyanide and with Murakami's reagent, and that the susceptibility of the steel to intergranular corrosion be determined by the acidified copper sulfate solution test. Experiments along these lines were carried out by the different cooperating laboratories, and in addition one of the laboratories also employed X-ray diffraction tests to obtain additional information concerning the precipitate at the grain boundaries of the steel when heated at such temperatures. The results of these tests are also included in the form of an appendix to this report. It seemed necessary to pursue the metallographic study of the austenitic chromium-nickel steels to determine the degree to which different laboratories could secure comparable results with similar techniques, and to determine the type of information concerning the properties of the steel that would be supplied by microscopic examination. It was expected in this way to establish the respective values of these methods of examination.

The analysis of the steel representing heat No. 1 was as follows:

Carbon, per cent	0.065
Manganese, per cent	0.43
Silicon, per cent	0.40
Chromium, per cent	18.15
Nickel, per cent	8.89

The samples of the steel had been annealed and held for one week at temperatures of 550, 650, 750, and 850 C., respectively. The cooperating metallographers were:

LABORATORY CODE LETTER	
J	J. W. Carey and H. R. Copson, International Nickel Company Research Laboratories
D	W. R. Angell, U. S. Navy
T	W. D. Forgeng, Union Carbide and Carbon Research Laboratories, Inc.
R	H. S. Rawdon, National Bureau of Standards
S	J. R. Vilella, U. S. Steel Research Laboratories

Etching and Preparation of Photomicrographs:

Each sample was prepared for metallographic examination by the cooperating laboratories and photographed at a magnification of 750 diameters. The cyanide etching was performed in a 10 per cent aqueous solution of sodium cyanide, and the current was applied for 3 to 10 min. After these photomicrographs were taken, the specimens were repolished and etched for 10 to 25 min. in a hot solution of Murakami's reagent (10 g. of potassium ferricyanide, 10 g. of potassium or sodium hydroxide, and 100 ml. of water).

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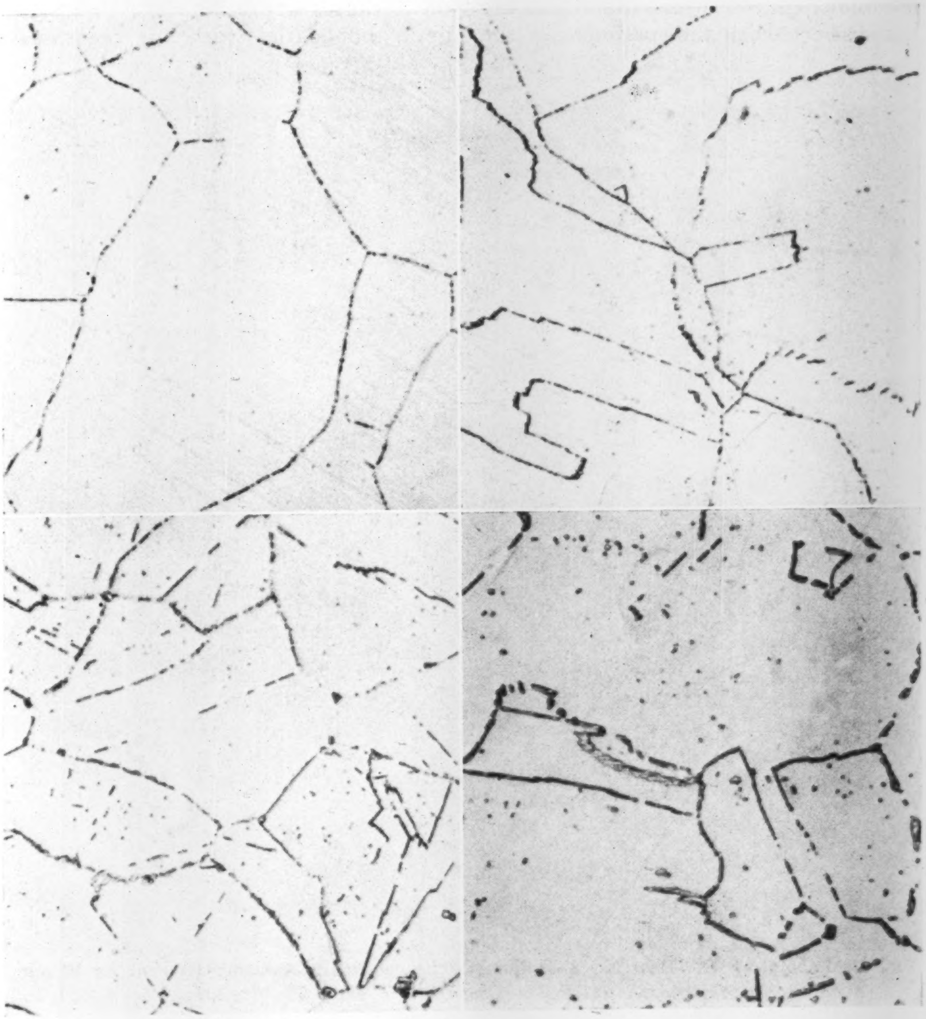
[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 203 (1939).


550 C. 650 C.

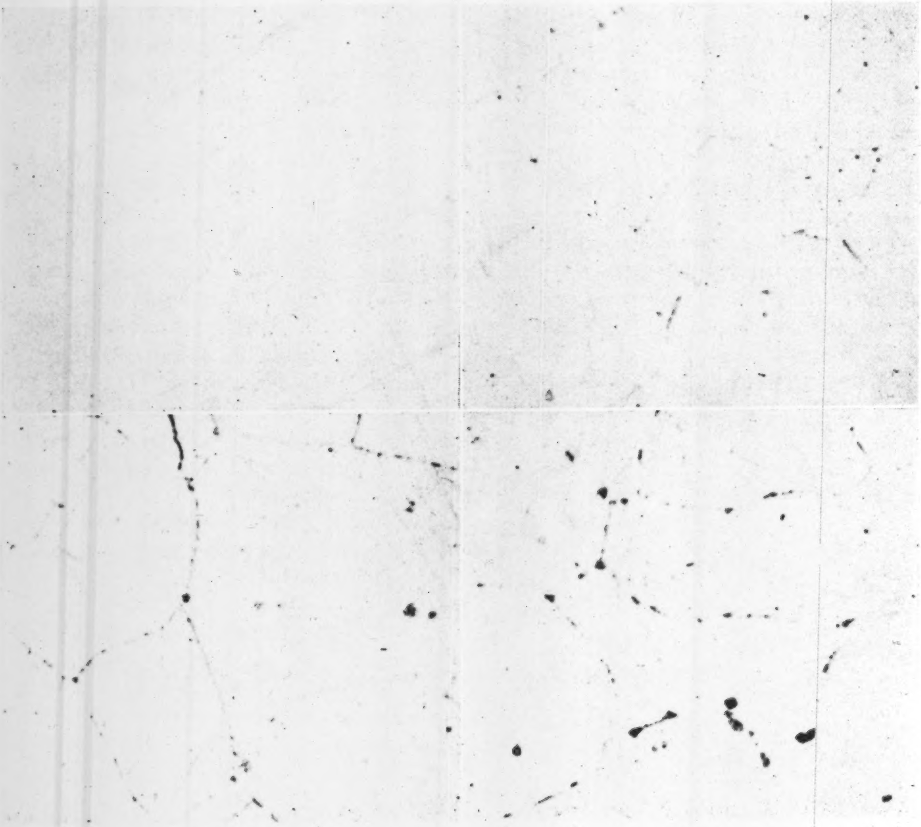
750 C. 850 C.

FIG. 1.—Laboratory D. Heat No. 1 Material Etched Electrolytically in Sodium Cyanide (10 per cent) for 3 min. (× 750).

The photomicrographs received from

four of the cooperating laboratories are shown in the attached figures. In view of variations in polishing, etching, and photographic technique, certain differences appear in the reproductions of the photomicrographs, which differences are less distinct when the specimens or the original prints are viewed directly; these differences were carefully considered before reaching the conclusion that the results obtained by the several laboratories were largely in agreement,—sufficiently so to establish the conclusions appearing in this report.

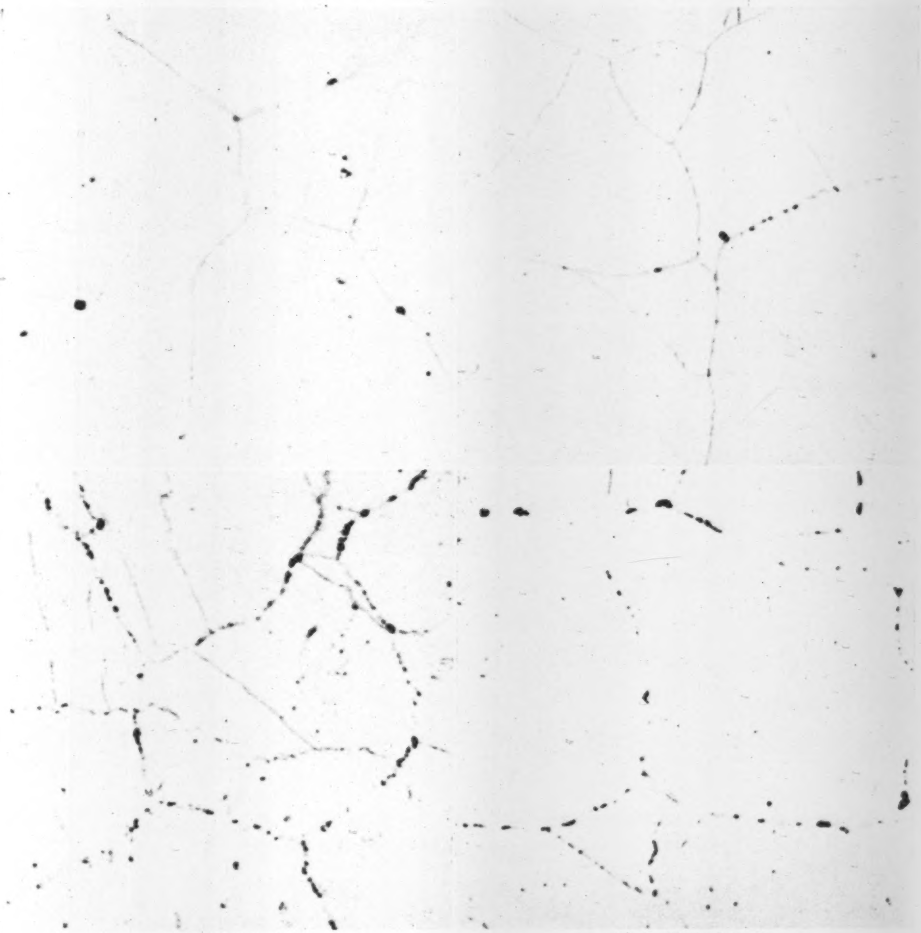
550 C. 650 C.



750 C. 850 C.

FIG. 2.—Laboratory D. Heat No. 1 Material Etched in Hot Murakami's Reagent for 10 min. (× 750).

The precipitate is darkened with the sodium cyanide etch and the austenitic grain boundaries are outlined in the usual etching time only when a precipitate is present. The 550 C. treatment causes a precipitation along the grain boundaries with an occasional discontinuity. After treatment at 650 C., the precipitated particles are slightly coarser and in some areas the precipitate occurs at the twinning planes. The precipitated particles are much larger and the grains heavily outlined after heating at 750 C., and precipitation along the twinning planes is quite general. After the treatment at 850 C., the structure is quite similar to that of the 750 C. specimen.



550 C., Etched 6 min.

650 C., Etched 6 min.

750 C., Etched 3 min.

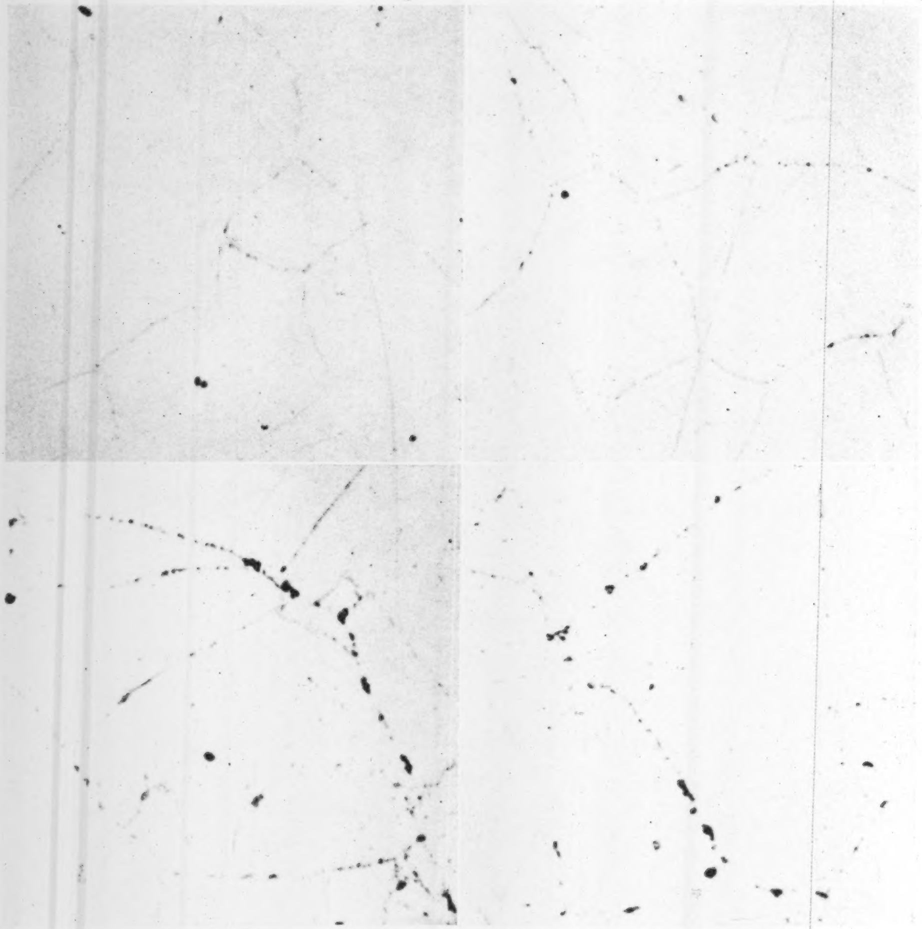
850 C., Etched 3 min.

FIG. 3.—Laboratory J. Heat No. 1 Material After 6 Days, Etched Electrolytically in Sodium Cyanide (10 per cent) (× 750).

Murakami's etch reveals only a trace of grain boundary material at 550 C. However, precipitated particles may be noted at close intervals along the grain boundaries of the specimen treated at 650 C. The particles become larger and more continuous at 750 C., and in this specimen precipitation may be noted at the twinning planes. At 850 C., the particles are slightly larger but they lack the continuity of those noted in the specimen heated at 750 C.

Embrittlement Tests:

The embrittlement tests were made by exposing identical samples of the steel from heat No. 1 for a period of 100 hr. to a boiling acidified copper sulfate solution containing 47 ml. per liter of sulfuric acid (sp. gr. 1.84) and 13 g. per liter of copper sulfate, using a reflux condenser. The relative degree of susceptibility was determined by visual examination of the outer surfaces of the specimens after bending, by the angle through which the specimens could be bent before intergranular cracks were observed, by hammering a corner of a cube (laboratory J only), and by weight loss.

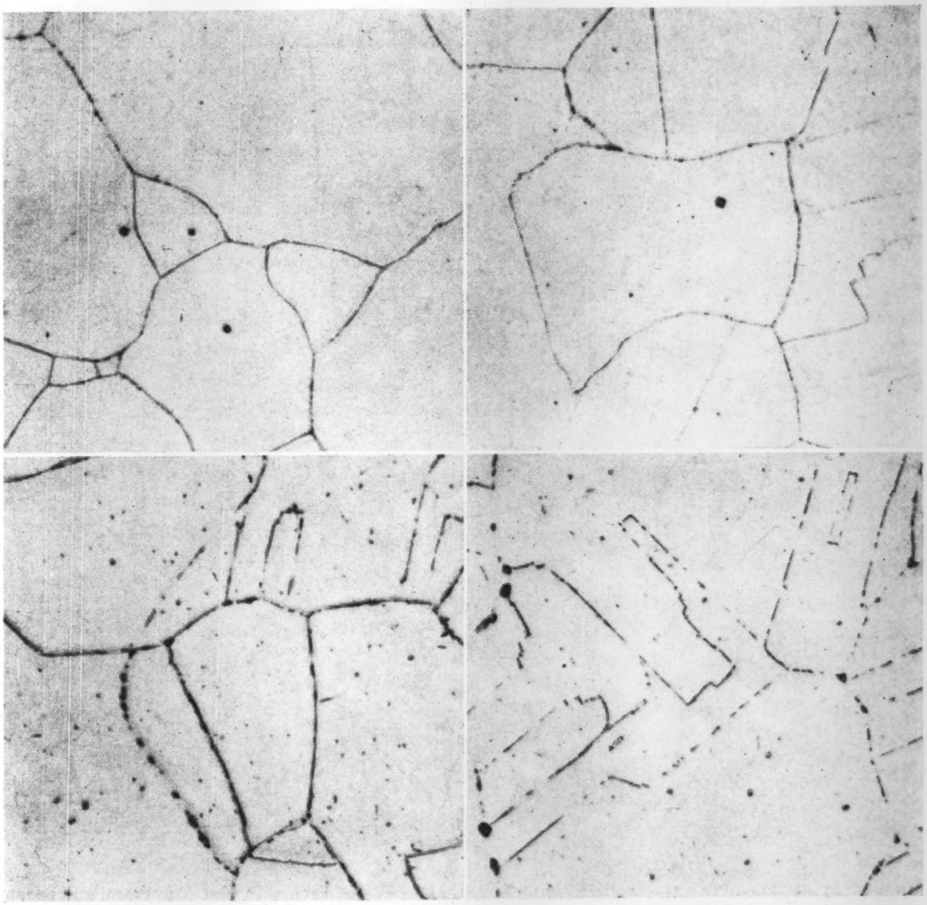

550 C., Etched 25 min.

650 C., Etched 15 min

750 C., Etched 15 min.

850 C., Etched 15 min.

FIG. 4.—Laboratory J. Heat No. 1 Material After 6 Days, Etched in Hot Murakami's Reagent (× 750).



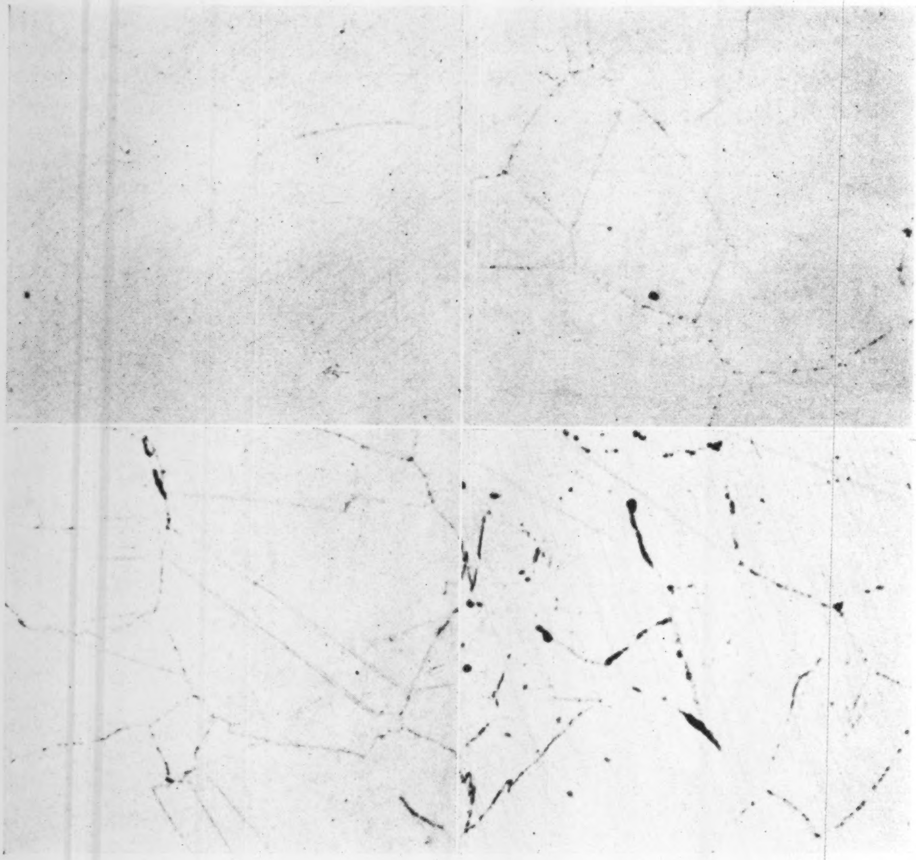
550 C. 650 C.

750 C. 850 C.

Fig. 5.—Laboratory T. Heat No. 1 Material After One Week Etched Electrolytically in Sodium Cyanide (10 per cent) for 3 min. (× 750).

The results of the embrittlement tests conducted by laboratory D are as follows:

Specimen Heated at	
550 C.	Failed badly. Angle of bend approximately 22 deg. when intergranular cracks were first observed.
650 C.	Failed badly. Angle of bend approximately 25 deg. when intergranular cracks were first observed.
750 C.	Failed. Angle of bend approximately 80 deg. when intergranular cracks were first observed.
850 C.	No failure. Angle of bend approximately 180 deg. without showing intergranular cracks.



550 C. 650 C.

750 C. 850 C.

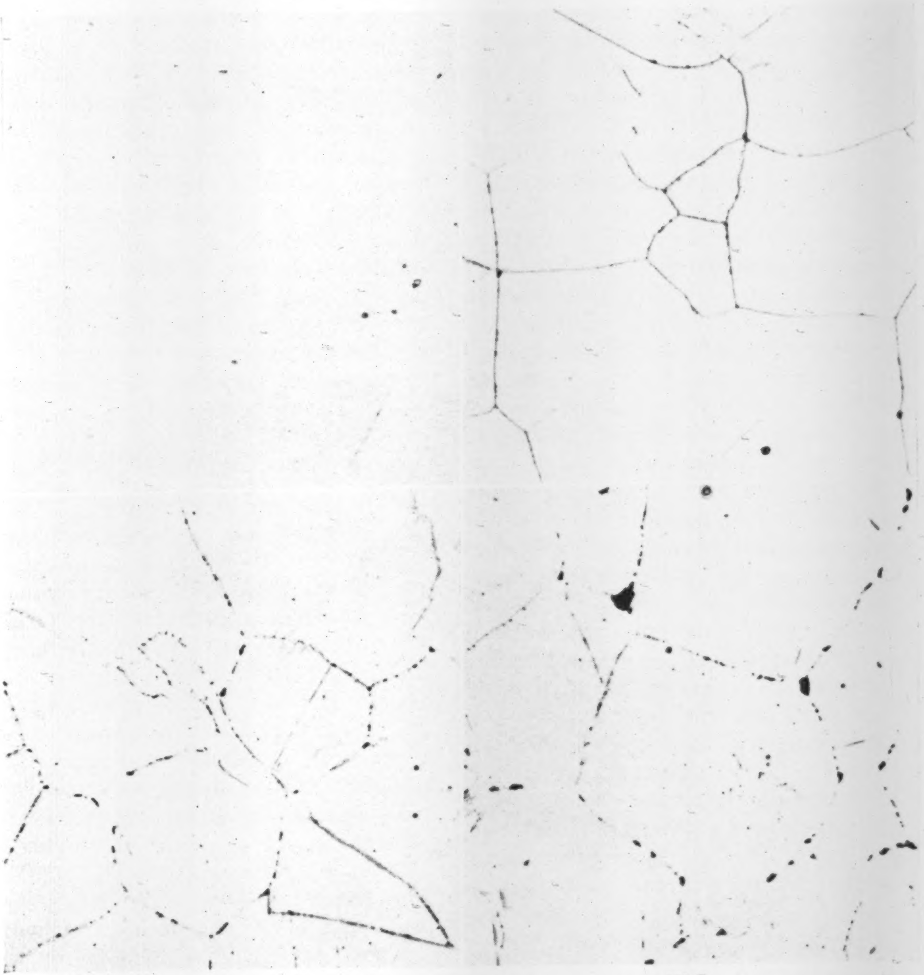
FIG. 6.—Laboratory T. Heat No. 1 Material After One Week, Etched in Hot Murakami's Reagent for 15 min. (× 750).

The results of similar tests reported on a weight-loss basis by laboratory J are as follows:

SPECIMEN FROM HEAT NO. 1 HEATED AT	WEIGHT LOSS IN 100-HR. STRAUSS TEST	
	G.	MG. PER DM. PER DAY
550 C..........	0.1558	470
650 C..........	0.0194	45
750 C..........	0.0007	2
850 C..........	0.0009	2

Laboratory J stated that upon removal from the boiling acidified copper sulfate solution the specimen heated at 550 C. was partially covered with crystals of copper and its polished surface showed granulation. Hammering one corner produced some cracking and crumbling. The specimen heated at 650 C. also showed slight granulation of its polished surface and hammering one corner produced some cracking, while the specimens heated at 750 C. and 850 C seemed unaffected.

The data submitted by laboratory T showed that after heating at 550 and 650 C. the steel exhibited intergranular attack on treatment in the boiling copper sulfate solution. The specimen heated at 750 C. showed slight cracking on bending, which indicated very mild susceptibility to intergranular corrosion, but the specimen heated at 850 C. exhibited no signs of attack at the grain boundaries.



550 C. 650 C. 750 C. 850 C.

FIG. 7.—Laboratory R. Heat No. 1 Material Etched Electrolytically in Sodium Cyanide (10 per cent) for 10 min. (× 750).

While all of the results from each laboratory for each heat treatment are not absolutely identical, there is sufficient similarity to permit the statement that the effects of the two lower heat-treating temperatures are quite similar and, likewise, the effects of the two higher heat-treating temperatures are quite similar. Between 650 and 750 C. a rather large difference in etching and corrosion behavior has occurred. At the lower temperatures the steel is more susceptible to attack in the Strauss test, and the precipitation revealed by the etching reagents is fine and more nearly continuous in character.

The Murakami etch, when applied to the specimens heat-treated at 550 C., does not behave similarly in the hands of all of the investigators (in two cases out of four, no intergranular developments appear), and this is extremely important because this appears to be the

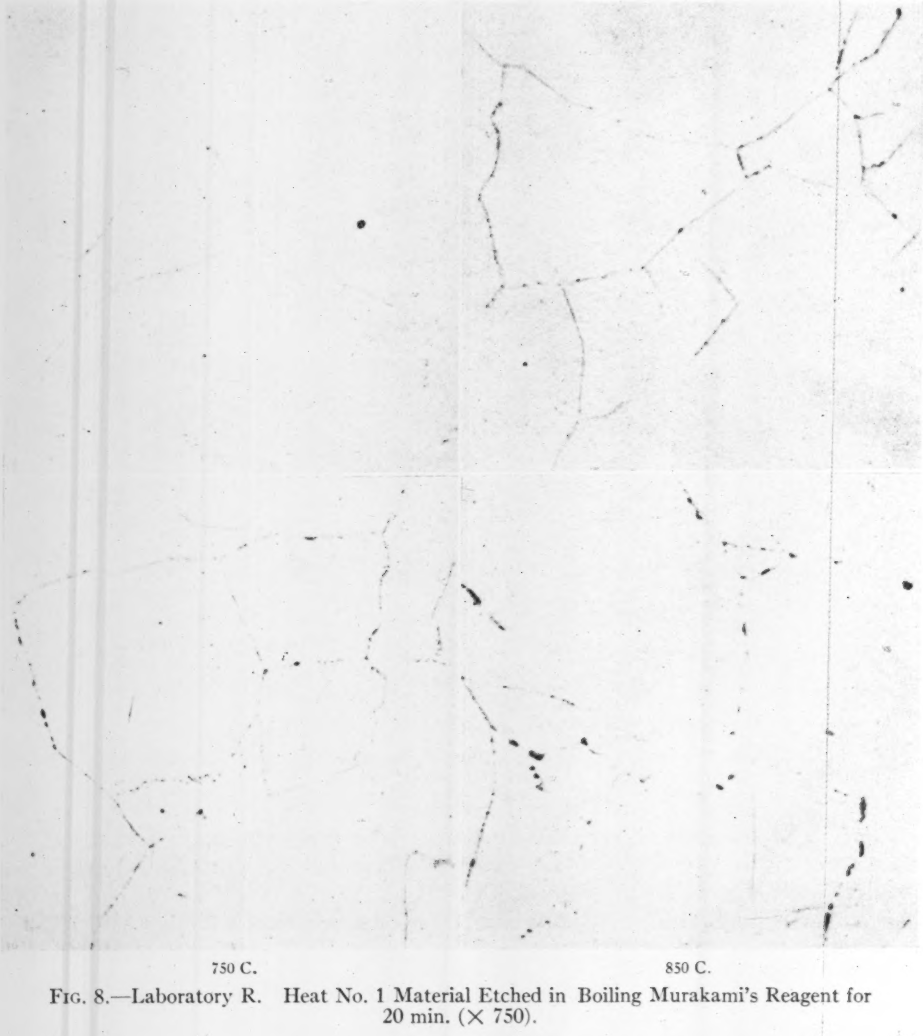


FIG. 8.—Laboratory R. Heat No. 1 Material Etched in Boiling Murakami's Reagent for 20 min. (× 750).

temperature of maximum susceptibility to intergranular attack. On the other hand, at the higher temperatures the results obtained by the several investigators are quite similar.

The sodium cyanide etch also is not perfectly satisfactory, for, although it indicates in the hands of each operative a definite condition at the grain boundaries, there appears to be considerable difference in the severity of the attack that is not disclosed by the time element alone. The reliability of its indications of susceptibility is, however, much better than that secured by the Murakami reagent. Further study of the etching conditions appears to be indicated if either or both reagents are to be employed with assurance of like interpretation by different observers.

Summary:

It is apparent from the photomicrographs obtained in the study of the structure of heat No. 1 that sodium cyanide is more positive and reliable than Murakami's reagent for detecting precipitated carbides (as shown by the data of the appendix) in the low-carbon 18 per cent chromium, 8 per cent nickel steels. However, neither reagent is instrumental in revealing a difference in microstructure commensurate with the relative susceptibility to intergranular corrosion, at least, so far as this is indicated by the Strauss test.

Although the results of these or the previous experiments[1] have not been instrumental in fulfilling the original purpose of the study, that is, to determine the nature of the material precipitated at the grain boundaries when austenitic stainless steel is subjected to heat treatment that renders it susceptible to intergranular corrosion, they have established that there is no correlation between the amount of precipitated material observed in the grain boundaries by microexamination and the susceptibility to intergranular corrosion.

There is some indication that when the intergranular precipitation is thin and continuous, intergranular corrosion susceptibility is high, and when the separated particles between the grains are large, distinct, and not continuous, the susceptibility is very low or absent. This statement, however, cannot be established as positive without appreciable further work, which should be accompanied by efforts to arrive at a uniform and acceptable etching procedure capable of producing uniform results by any skilled metallographer, and also an attempt to determine whether any etching procedure can adequately predict susceptibility to intergranular corrosion. In X-ray studies by one of the laboratories, as reported in the appendix, the presence of the chromium carbide Cr_4C was noted in all specimens. It is pertinent to note that the sample heated at 550 C., which showed the greatest susceptibility to attack, contained the least amount of chromium carbide.

Respectfully submitted on behalf of the subcommittee,

RUSSELL FRANKS,
Chairman.

APPENDIX

X-ray diffraction patterns were taken of residues from each specimen by laboratory T. The residues were obtained by treatment of the samples with a solution of hydrochloric acid and potassium copper chloride. It was found that the residue from the 850 C. specimen was well crystallized Cr_4C with traces of Cr_2N and austenite. The residue from the 750 C. specimen was a mixture of Cr_4C and austenite. The amount of Cr_4C in the residue from the 650 C specimen was much lower than in the 750 C. specimen, and only a trace was present in the residue from the 550 C. specimen. The bulk of the residue in the latter samples was austenite.

REPORT OF THE RESEARCH COMMITTEE

ON

FATIGUE OF METALS

Owing to the demands of war conditions, it has not been found feasible to hold a formal meeting of the Research Committee on Fatigue of Metals during this year. The cooperative arrangement with *Metals and Alloys* has been continued whereby this committee furnishes its services in the abstracting of articles on fatigue of metals for publication in the Metallurgical Engineering Digest of this Journal.

The committee, acting on a suggestion from G. F. Jenks, chairman of Committee E-9 on Research, is considering suggestions for special problems directly connected with the war effort in the study of which the committee could cooperate. Arrangements are being made for the committee to have close contact with one such investigation which is just being started.

During the year the committee has received additional allotments from the A.S.T.M. Research Fund for the purpose of carrying on the Study of the Effect of Size of Specimens on the Fatigue Strength of Metals. The committee was able to obtain a sufficient amount of three types of steel, and a progress report of this investigation is presented as an Appendix to this report.

This report has been submitted to letter ballot of the committee which consists of 21 members; 15 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. F. MOORE,
Chairman.

APPENDIX

PROGRESS REPORT ON THE EFFECT OF SIZE OF SPECIMEN ON FATIGUE STRENGTH OF THREE TYPES OF STEEL

PREPARED BY H. F. MOORE[1] AND D. MORKOVIN[2]

SYNOPSIS

Tests have been carried on at the Talbot Materials Testing Laboratory at the University of Illinois under the auspices of the A.S.T.M. Research Committee on Fatigue of Metals on specimens of two plain carbon steels (S.A.E. 1020 and S.A.E. 1035) and one heat-treated chromium-molybdenum steel (S.A.E. X4130). Five different sizes of specimens were tested for each kind of steel; with minimum test diameters of $\frac{1}{8}$, $\frac{1}{4}$, $\frac{1}{2}$, $\frac{7}{8}$ or 1 in., and $1\frac{1}{2}$ or $1\frac{3}{4}$, or 2 in., respectively. The specimens were cut from bars 3 or $3\frac{1}{2}$ in. in diameter, and care was taken to have the most stressed fiber of each specimen as nearly as possible the same distance from the axis of the bar. Surveys of the Brinell hardness of the cross-section of the bars were made to serve as a basis for correction of slight variations in this distance. In machining, care was taken to avoid overheating.

All fatigue tests were made in rotating-cantilever testing machines. The results of the tests showed a maximum size effect of about 30 per cent of the endurance limit for 2-in. specimens of the S.A.E. 1020 steel. There was also shown a tendency for the endurance limit to become constant for specimens $\frac{1}{2}$ in. minimum diameter and larger.

A comparison of these results with published results of other "size effect" studies was made, and a diagram is included showing the field of "scatter" of the results found in the various investigations.

Scope of Proposed Investigation Submitted:

The investigation of "size effect" on fatigue strength was planned to cover at least three main lines of study: (*1*) Effect of size on unnotched, polished specimens, (*2*) Effect of size on notched specimens, and (*3*) Effect of internal stresses in metals studied. This progress report has to do with the first of these objectives. Work on the second has been begun and work on the third is under consideration.

Cooperating Laboratories:

In carrying out these tests the Westinghouse Research Laboratories, the Massachusetts Institute of Technology Materials Laboratory, the Kansas State College Shops, and the University of Wisconsin, either are machining specimens, making tests, or have agreed to do so upon receipt of specimens. This particular report covers tests made during the past year at the University of Illinois Materials Testing Laboratory where there was equipment for testing specimens ranging in size from $\frac{1}{8}$ to 2 in. in diameter.

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[1] Research Professor of Engineering Materials, University of Illinois, Urbana, Ill.

[2] Instructor in Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

Metals Tested:

For the first lot of metal for the investigation the following types of steel were chosen: (*1*) An S.A.E. 1020 carbon steel, hot rolled and furnished by the Carnegie-Illinois Steel Corp., (*2*) An S.A.E. 1035 steel, hot rolled and also furnished by the Carnegie-Illinois Steel Corp., and (*3*) An S.A.E. X4130 chromium-molybdenum steel, heat treated to a Brinell hardness of approximately 300 and furnished by the Timken Roller Bearing Co.

The S.A.E. 1020 steel comprised 15 bars, each 20 ft. long and $3\frac{1}{2}$ in. in diameter. These were all from the same heat of steel. The S.A.E. 1035 steel comprised 16 round rods each 19 ft. long and $3\frac{1}{2}$ in. in diameter. The S.A.E. X4130 steel comprised 11 pieces of round rod in lengths varying from 17 to 21 ft. long and 3 in. in diameter.

TABLE I.—HEAT ANALYSES OF THE STEELS USED IN SIZE EFFECT TESTS.

Steel	Chemical Content, per cent						
	Carbon	Manganese	Silicon	Chromium	Molybdenum	Sulfur	Phosphorus
S.A.E. 1020	0.22	0.46	0.20	...	...	0.033	0.014
S.A.E. 1035	0.34	0.78	0.24	...	...	0.028	0.016
S.A.E. X4130	0.31	0.55	...	1.08	0.20	0.019	0.017

Properties of the Metals Tested:

Table I shows the chemical analyses of the three steels furnished and Table II gives the mechanical properties.

Fatigue Specimens:

All of the fatigue specimens were rotating-beam specimens tested in rotating-cantilever machines. The largest specimen for the S.A.E. 1020 steel was 2 in. at the minimum diameter, for the 1035 steel, $1\frac{3}{4}$ in. at the minimum diameter, and for the chromium-molybdenum X4130 steel, $1\frac{1}{2}$ in. at the minimum diameter. These different sizes were made necessary by the limit of allowable load on the fatigue testing machine used for these specimens. The second largest specimens were 1 in. in minimum diameter for the 1020 and 1035 steels, and $\frac{7}{8}$ in. in minimum diameter for the X4130 steel. The third largest lot of specimens were $\frac{1}{2}$ in. in minimum diameter, the fourth largest lot of specimens were $\frac{1}{4}$ in. in minimum diameter, and the smallest specimens were $\frac{1}{8}$ in. in minimum diameter.

TABLE II.—MECHANICAL PROPERTIES OF THE STEELS USED IN SIZE EFFECT TESTS.

Steel	Yield Strength, psi.	Tensile Strength, psi.	Elongation in 2 in., per cent	Reduction of Area, per cent	Average Brinell Hardness Number
S.A.E. 1020	32 300[a]	62 000	28.0	59	125
S.A.E. 1035	47 200[a]	87 600	25.0	50	175
S.A.E. X4130	115 600[b]	141 800	17.0	60	300

[a] Yield point by halt of pointer, rate of strain below yield point 0.042 per cent per min.

[b] Yield strength by offset of 0.2 per cent, rate of strain below yield strength 0.042 per cent per min.

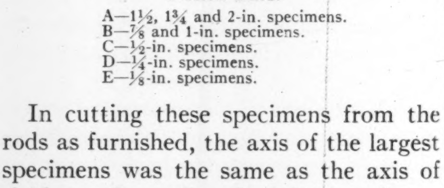

FIG. 1.—Location of the Test Specimens in the Rolled Bars.

A—1½, 1¾ and 2-in. specimens.
B—⅞ and 1-in. specimens.
C—½-in. specimens.
D—¼-in. specimens.
E—⅛-in. specimens.

In cutting these specimens from the rods as furnished, the axis of the largest specimens was the same as the axis of the bar. In cutting the other specimens from the bars, it was attempted to bring the inner end of the minimum diameter as nearly as possible to the same radius of the axis of the bar as was the surface of the largest specimens. The location of the specimens in the bars is shown in Fig. 1. It was not found feasible to cut all the specimens at precisely the radius desired, and allowance for the change of fatigue strength of the metals was made on the basis of a survey of Brinell hardness. Figure 2 shows the piece cut from each test bar about 3 ft. from the end, on which Brinell hardness measurements were taken as shown in the figure.

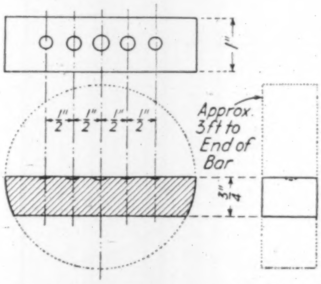

FIG. 2.—Location of Specimens for Brinell Hardness Survey of Rolled Bar.

Small circles show the location of the indentations.

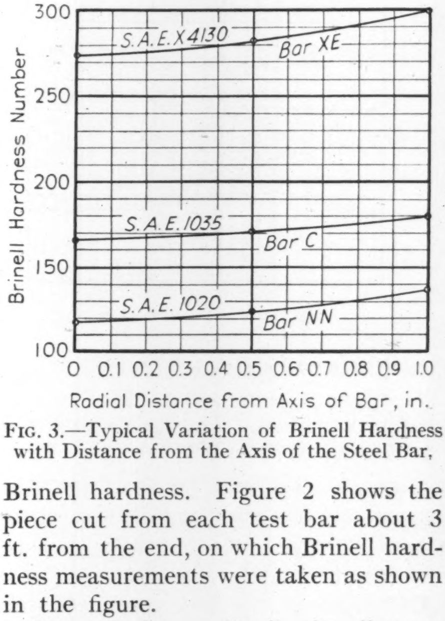

FIG. 3.—Typical Variation of Brinell Hardness with Distance from the Axis of the Steel Bar.

Figure 3 shows a distribution diagram of hardness at different radial distances from the axis of typical bars. All the bars showed stronger material 1 in. from the axis than at points nearer the axis. In Fig. 3 values for the points at distances of 0.5 in. and 1 in., respectively, from the axis are the average of two determinations. The results reported for each specimen tested in fatigue have been adjusted to the approximate average hardness for each metal as follows:

S.A.E. Steel	Brinell Hardness Number
1020	125
1035	175
X4130	300

It was assumed that over the narrow range of Brinell hardness the endurance limit for the steel would be directly proportional to that hardness.

The general type of fatigue specimen is shown in the diagram in Fig. 4. The reduced section was turned down with a tool swung on a radius equal to very nearly five times the minimum diameter of the specimens ($R = 5d$.) In this type of specimen the maximum stress is not quite at the minimum section, since for a short distance from the minimum section the increase in the moment arm l overbalances the increase in the diameter of the specimen. The maximum stress is found at some section AB. This excess of maximum stress over the stress at the minimum section was determined for each of the sizes of specimens tested. It was found to be about 2.6 per cent for the 2-in. specimens and was negligible for the $\frac{1}{2}$, $\frac{1}{4}$ and $\frac{1}{8}$-in. specimens. The actual fatigue failures

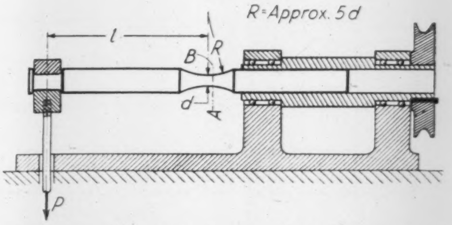

FIG. 4.—Diagram of Typical Test Specimen and Rotating-Cantilever Fatigue Machine.

occurred at sections in the vicinity of the sections of the maximum stress.

In machining the fatigue specimens from the bars, care was taken to avoid overheating. This was done by using light cuts, slow cutting speeds, by cutting under a stream of coolant, or by a combination of these methods.

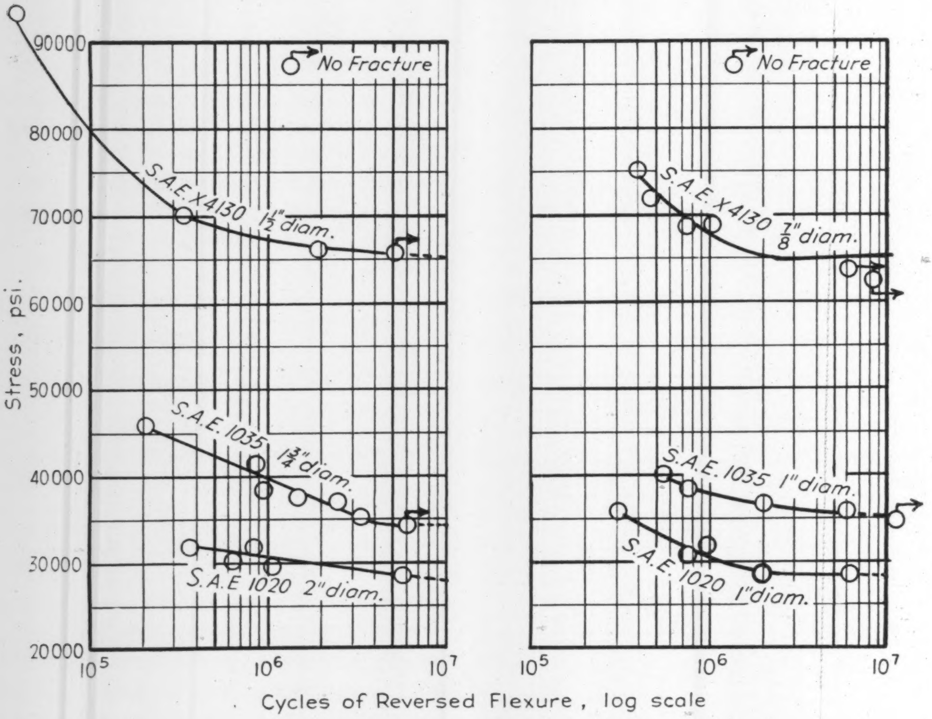

FIG. 5.—*S-N* Diagrams for $\frac{7}{8}$, 1, $1\frac{1}{2}$, $1\frac{3}{4}$, and 2-in. Specimens.

In polishing each specimen, it was rotated slowly about its axis while a rapidly rotating round bar wrapped with emery cloth was held with its axis at an angle to the axis of the specimen. The resulting "polishing scratches" were approximately at 45 deg. with the specimen axis.

Testing Machines:

The testing machines were all of the rotating-cantilever type shown in the diagram in Fig. 4. The machines were fitted with ball bearings, including the bearings in the load hanger bearing. The load hanger was also pivoted about a horizontal diametral axis. In the tests of the larger specimens, on account of the rise in temperature, it was found necessary to reduce the speed of the testing machines. The speeds used were as follows: For the 2, $1\frac{3}{4}$, and $1\frac{1}{2}$-in. specimens, 400 rpm.; for the 1 and $\frac{7}{8}$-in. specimens, 800 rpm.; for the $\frac{1}{2}$-in. specimens, 1200 rpm.; for the $\frac{1}{4}$-in. specimens, 2400 rpm.; and for the $\frac{1}{8}$-in. specimens 3200 rpm. A few $\frac{1}{8}$-in. specimens were tested at 6000 rpm. and the results of these fitted into the same *S-N* diagrams as were found for the tests at 3200 rpm. By thus adjusting the speeds, the surface temperature of the specimens at the critical sections was kept below 110 F.

Test Results:

Figures 5 to 7, inclusive, show *S-N* diagrams. In these diagrams each test result is represented by a circle whose radius represents 1000 psi. on the stress scale. This same plan is followed through the test diagrams. These tests are not quite complete, but it is believed that endurance limits may be estimated with sufficient accuracy to make this report worth while.

Figure 8 shows a summary of these 1942 A.S.T.M. size effect tests performed by the authors at the Talbot Laboratory of the University of Illinois. It may be observed that all three curves, for the three different steels, show a tendency to flatten out for specimens of diameters $\frac{1}{2}$ in. and larger. For the sake of shortness and definiteness, the constant ordinate of the horizontal portion of each of the curves in Fig. 8 for the three different steels will be referred to in this report as "endurance limit of large specimens" of that particular steel.

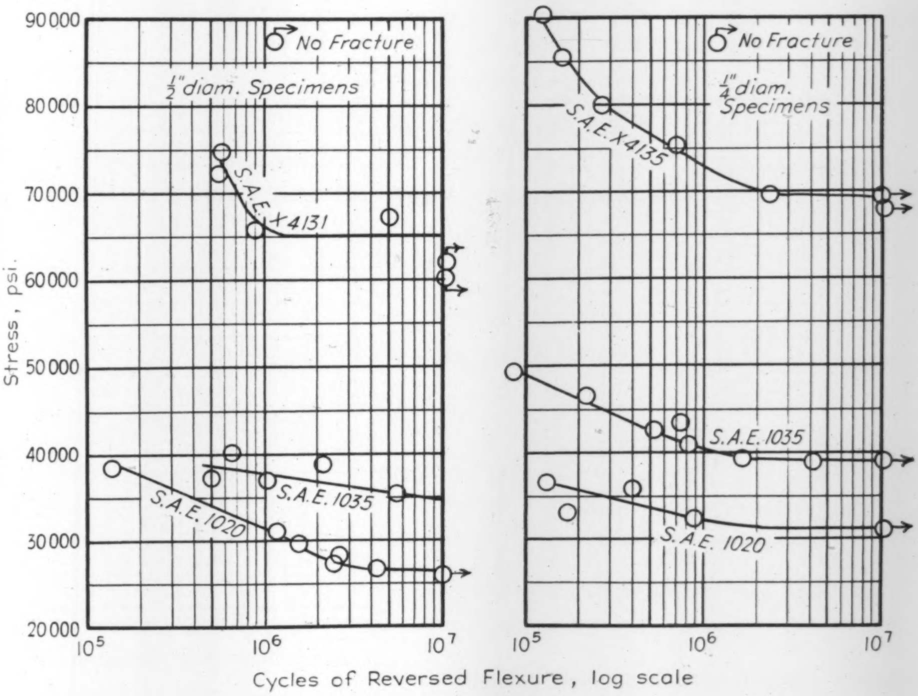

FIG. 6.—*S-N* Diagrams for $\frac{1}{4}$ and $\frac{1}{2}$-in. Specimens.

Thus the tests performed so far in the 1942 A.S.T.M. size effect series seem to indicate that the endurance limit decreases with increasing size, but tends to approach a lower limit, the "endurance limit of large specimens." Beyond a certain size, there seemed to be no indication of any further drop in endurance limit with further increase in size.

Mailänder and Bauersfeld[3] in the discussion of their results (size effect in reversed torsion), express a similar conclusion. In their case, the flattened-out portion of the endurance limit-size curve was not yet reached by the polished, solid torsion specimen of the largest size tested (1.77 in. diameter). However, a distinct tendency to flattening out was shown by the endurance limit-size curves for their polished tubular torsion specimens and solid polished torsion specimens with fillets, keyways, and cross-holes.

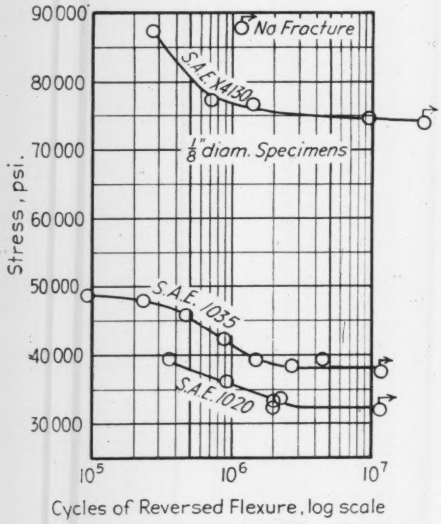

FIG. 7.—*S-N* Diagrams for $\frac{1}{8}$-in. Specimens.

The extent and form of the critical regions and the states of stress are different in differently shaped specimens. This may explain the observation that the properly drawn endurance limit-size curves through the actual test result points of the various Mailänder-Bauersfeld test series cross and show flattening out at different sizes.[4] For the same reasons one should not expect to find the flattening out of endurance limit-size curves for torsion tests at the same specimen sizes as for the rotating-cantilever-bending specimens.

The control data in the investigation of H. F. Moore and R. L. Jordan furnish two sets of data from which endurance limit-size curves can be drawn for polished rotating-cantilever specimens in reversed bending.[5] That investigation was not intended to emphasize size effect, and therefore the location of the specimens in the bar

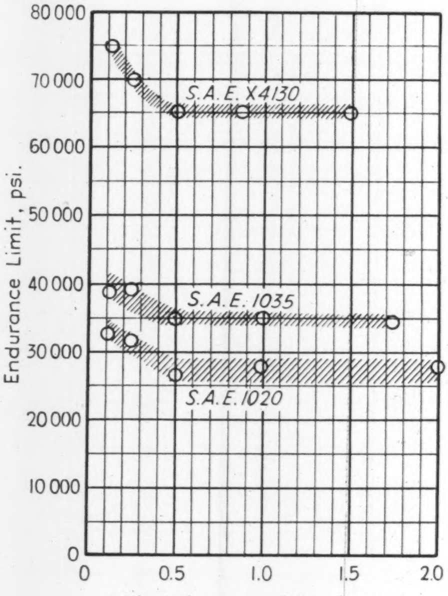

FIG. 8.—Endurance Limit—Size Diagrams.

Shaded area represents the estimated "scatter" area due to experimental error.

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[3] Mailänder and Bauersfeld, "Einfluss der Probengrösse und Probenform auf die Dreh-Schwingungfestigkeit von Stahl," *Technische Mitteilungen Krupp*, Year 2, No. 5, December, 1934.

[4] *Loc. cit.*, p. 149—"Besides that, the curves were somewhat evened out with respect to each other, so that there is no mutual crossing." The above-mentioned flattening-out is more evident if the curves are redrawn through the actual points without arbitrary adjustment.

[5] H. F. Moore and R. L. Jordan, "Stress Concentration in Steel Shafts with Semi-circular Notches," *Proceedings*, Fifth Internat. Congress of Applied Mechanics, pp. 188–192 (1938).

was not studied so carefully, as was done in this 1942 A.S.T.M. series. Neither was a Brinell hardness survey carried out and therefore the endurance limits of the specimens of various sizes are not quite comparable on the basis of identity of properties of the material. However, the series for S.A.E. 1020 steel shows clearly a flattening out, whereas the results for S.A.E. 2345 steel show too much scatter to reach a conclusion. However, all the test series with notched specimens show flattening out of endurance limit-size curves.

The Appendix to the 1941 Report[6] of the A.S.T.M. Research Committee on Fatigue of Metals contains two endurance limit-size curves for steels. Neither curve shows data for specimens of as large sizes as those sizes at which similar curves in this 1942 investigation show a flattening out. The 1941 series will be hereafter referred to in this paper as the 1941 A.S.T.M. series, and the present investigation as the 1942 A.S.T.M. series.

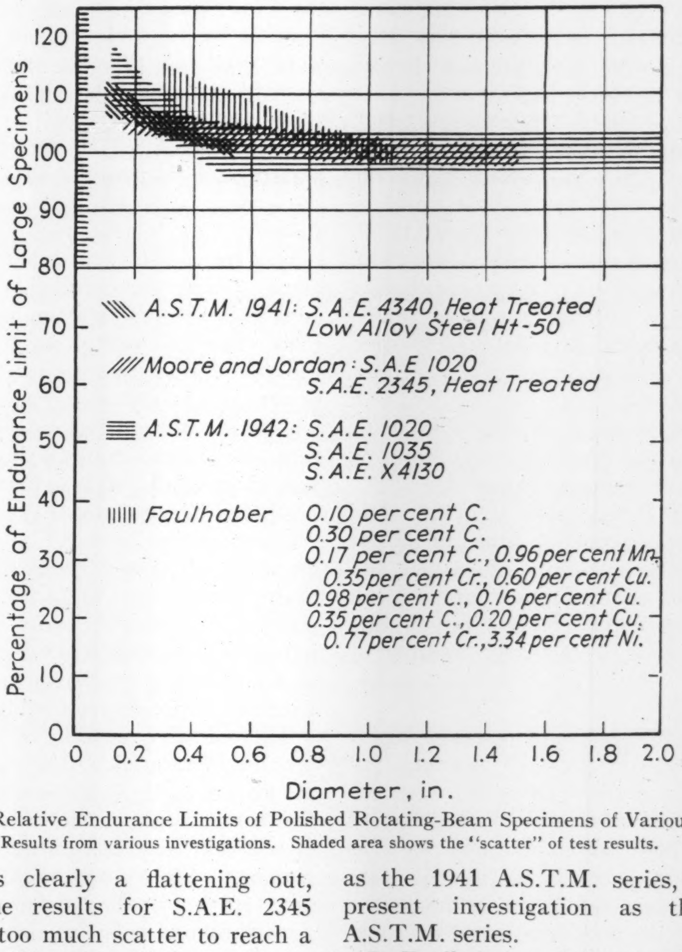

Fig. 9.—Relative Endurance Limits of Polished Rotating-Beam Specimens of Various Sizes.
Results from various investigations. Shaded area shows the "scatter" of test results.

R. Faulhaber carried out an investigation of size effect in reversed bending (constant bending moment Schenck type machine) for six different steels, with polished specimens, ground specimens, specimens merely turned, and specimens with three various depths of circumferential 90 deg. V-notches.[7] For two of the steels tested, the flattening out of the endurance limit-size curves was not reached even with the largest size of the polished specimen (1.075 in. diameter). For the other steels, the data are not sufficient to determine this. However, a distinct flattening out of endurance limit-size curves may be observed even with the sizes tested in the case of notched specimens.

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[6] *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 133 (1941).

It is not proposed to discuss size effect in notched specimens in the present progress report. Size effect tests on notched specimens are now in progress at the Materials Testing Laboratory of the University of Illinois. Notwithstanding the results of investigations on size effect in notched specimens mentioned above, the data are still too few to justify drawing conclusions in this progress report.

The authors know of only one set of published data on endurance limits of specimens of diameters larger than 2 in., —those of Horger and Neifert.[8] The following is a quotation from the paper: "No discussion of size effect and its influence on the endurance limit for the various size specimens is given in this paper because of the large number of variables, which preclude its evaluation."

The differently shaded areas in Fig. 9 are the "scatter regions" in which fall the points corresponding to the endurance limits of many varieties of steels. These were obtained by the investigators listed in the figure. Also the steels used are listed in the figure. All the results are for polished rotating-beam specimens in bending. Faulhaber's results are for constant bending moment specimens, the rest mostly for cantilever type specimens. The "endurance limit of large specimens" for each steel was used as 100 per cent and the endurance limits of smaller specimens were expressed as percentages on this basis. In Faulhaber's series and the 1941 A.S.T.M. series the mean endurance limits of the largest specimens were used as 100 per cent. Consequently, it might be that the corresponding "scatter regions" for Faulhaber and the 1941 A.S.T.M. series should be placed somewhat higher with respect to the main 1942 A.S.T.M. series than shown. The "scatter regions" were extended in height by allowing for experimental error. Proceeding in the above described way, less scatter was obtained than when the endurance limits of all the $\frac{1}{4}$-in. specimens were used as a basis of comparison (100 per cent). It is not felt that a straight-line extrapolation to zero size (as was done by Faulhaber) is justifiable at all. It is believed that the combined scatter region covers safely the endurance limits of all the steels listed for all sizes of polished rotating-beam bending specimens up to 2 in. in size. The Faulhaber series is probably not quite comparable to the rest because of the different type (constant bending moment) of specimens used by him.

The tentative meaning of the diagram may be illustrated by the following numerical example: Suppose it is desired to find a safe value of the endurance limit of a 2-in. diameter rotating-beam steel member. From a series of 0.3-in. specimens cut out of the bar from which the 2-in. specimen is to be machined, from the same depth as the surface of the 2-in. member would occupy, an endurance limit of, say, 40,000 psi. was obtained. The ordinate of the top margin of the scatter region at an abscissa of 0.3 in. is about 116 per cent. The ordinate of the bottom edge of the scatter region at 2 in. is 97 per cent. Therefore, a safe estimate, according to all the data collected up to the present, is that the endurance limit of the 2-in. polished specimen probably will not be below: $40,000 \times \frac{97}{116} = 33,400$ psi.

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[7] *Mitteilungen*, Forschungs-Institute der Vereingte Stahlwerke, Dortmund, Vol. 3, pp. 153–171 (1933); also *Stahl und Eisen*, Vol. 53, pp. 1106–1108 (1933).

[8] O. J. Horger and H. R. Neifert, "Fatigue Strength of Machined Forgings 6 to 7 in. in Diameter," *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 723 (1939).

The results of this 1942 A.S.T.M. investigation are probably directly applicable only to polished rotating steel members loaded as cantilever beams. Members with other surface finishes, with stress concentrations, and with other types of stresses have been shown to behave differently, at least so far as the magnitude of the size effect is concerned.[3,5,7]

It is not proposed to discuss possible causes of size effect in the present progress report. Consideration of such causes, including internal stresses in the rolled bars and test specimens, effect of "stress gradient," grain size, increased probability of occurrence of defects (due to external causes or inherent in the material itself) of critical size at the increased surface of large specimens, etc., as well as of the combination of several such probable causes, is in progress.

Acknowledgments:

Thanks are given to Messrs. G. F. Jenks and J. O. Leech for their effective aid in securing release of the steel needed for this investigation. Acknowledgments are made to the Timken Roller Bearing Co. for a special lot of steel furnished, and for a copy of its extensive data on fatigue tests of car axles and other large specimens. Acknowledgments are made to the Carnegie-Illinois Steel Corp. for special lots of steel furnished. Acknowledgments are also made to the cooperating laboratories named in the second paragraph of this paper.

The work in the Talbot Laboratory was carried on as a part of the regular work of the University of Illinois Engineering Experiment Station, Dean M. L. Enger, Director, and of the Department of Theoretical and Applied Mechanics, Professor F. B. Seely, Head.

REPORT OF JOINT RESEARCH COMMITTEE

ON

EFFECT OF TEMPERATURE ON THE PROPERTIES OF METALS

Progress Report to the Sponsor Societies

During the past year, the Joint Research Committee on Effect of Temperature on the Properties of Metals under the sponsorship of The American Society of Mechanical Engineers and the American Society for Testing Materials, held one meeting on December 2, 1941, at New York, N. Y. A second meeting of the committee will be held in June, 1942, in Atlantic City, N. J., prior to the presentation of this report at the annual meeting of the A.S.T.M. Several meetings of project committees have been held during the year.

Finances:

There appears in Table I a summary of the administration of funds during the yearly period of May 1, 1941, to April 30, 1942.

TABLE I.—SUMMARY OF RECEIPTS AND EXPENDITURES OF COMMITTEE FUNDS.

Balance as of April 30, 1941, shown in 1941 Report			$11 957.20
	RECEIPTS		
1941			
September	Interest on funds 10/1/40 to 9/30/41	$48.25	
November	The Engineering Foundation	250.00	
December	The Engineering Foundation	250.00	
1942			
March	The Engineering Foundation	250.00	
	Total Receipts		798.25
			$12 755.27
	EXPENDITURES		
1941			
June	Battelle Memorial Institute (Project No. 13)	$650.00	
June	Battelle Memorial Institute (Project No. 18)	618.71	
July	Battelle Memorial Institute (Project No. 18)	339.38	
October	Battelle Memorial Institute (Project No. 18)	658.49	
December	Battelle Memorial Institute (Project No. 18)	29.00	
	Total Expenditures		$2 295.58
Balance on hand, April 30, 1942			$10 459.69

Expenditures have been authorized and payments made to date on several research projects as follows:

	Maximum Expenditure Authorized	Payments Made
Project No. 10—Tubular Members Subject to Internal Pressures	$4 100.00	$3 500.00
Project No. 18—Effect of Variables on the High Temperature Properties of Metals (Part 3)	5 000.00	3 538.39
Project No. 25—Comparison of Short-Time Methods	1 000.00	500.00

Organization and Membership:

One new member was added to the committee in June, 1941, Mr. John S. Worth of the Bethlehem Steel Corp. Mr. Worth has long been active in the work of the committee, as a cooperator and adviser, associated with the late Mr. P. E. McKinney.

Research Projects:

There follows a summary of progress made in the various research projects during the year ending June, 1942.

Project No. 10 on Tubular Members Subject to Internal Pressures (H. J. Kerr, chairman).—The work covered by this project, carried out at the Massachusetts Institute of Technology, has now been completed, and it is anticipated that the report will be published in the near future by The American Society of Mechanical Engineers.

Project No. 16 on Relaxation (E. L. Robinson, chairman).—This project is inactive at present. It is planned to sponsor a technical session on this subject at a more convenient time.

Project No. 18 on Effect of Variables on the High-Temperature Properties of Metals. (H. W. Gillett, chairman).—Much of the creep equipment at Battelle Memorial Institute that has been utilized in the work of this project is now assigned to other important studies, and the completion of this project must be held till a later time.

Project No. 25 on Comparison of Short-Time Test Methods (J. S. Worth, chairman).—This project involves the comparison of results from a number of short-time test methods with those from long-time creep tests on two steels, the 0.35 per cent carbon steel K-20, and a carbon-molybdenum steel K-22. The creep test results for the steel K-20 are being taken from the many published results of tests on this steel; for the carbon-molybdenum steel K-22, creep tests at 850 F. and 1000 F. have been completed at the University of Michigan. A number of laboratories are cooperating in this work.

Despite the war conditions, the project committee has made real progress, and the report is now in course of preparation.

Project No. 26 on Test Methods (J. W. Bolton, chairman).—The committee has studied the Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials (E 21 – 37 T) and has recommended that this be changed to Recommended Practice for Short-Time Elevated Temperature Tension Tests of Metallic Materials.[1]

Respectfully submitted on behalf of the Joint Research Committee,

N. L. Mochel,
Chairman.

H. J. Kerr,
Vice-Chairman.

J. W. Bolton,
Secretary.

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[1] See Editorial Note, below.

Editorial Note

Subsequent to the annual meeting, the Joint Committee on Effect of Temperature on the Properties of Metals presented to the Society through Committee E-10 on Standards a revision of the Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials (E 21 – 37 T). The revised tentative standard was accepted by Committee E-10 on January 5, 1943, and appears in the 1942 Book of A.S.T.M. Standards, Part I, p. 1577.

REPORT OF COMMITTEE B-1
ON
COPPER AND COPPER-ALLOY WIRES FOR ELECTRICAL CONDUCTORS

Committee B-1 on Copper and Copper Alloy Wires for Electrical Conductors held three meetings during the year: in Chicago, Ill., on June 23, 1941, in Cleveland, Ohio, on March 3, 1942, and in New York, N. Y., on May 15, 1942.

The committee lost an active member of long standing in the death of H. A. Staples of the Phelps Dodge Copper Products Corp.

Several changes in membership have occurred during the year:

W. H. Bassett, Jr., Vice-Chairman of the committee, has entered the service as Captain in the Ordnance Division.

L. A. Ward, Chase Brass and Copper Co., Inc., new member.

M. A. Buell now represents the Bridgeport Brass Co., replacing R. S. Pratt.

P. A. Moody now represents the Western Electric Co., replacing C. H. Frank.

W. R. Hibbard, additional representative of The American Brass Co.

H. H. Stout, Jr., Phelps Dodge Copper Products Corp., new representative.

A. A. Jones, new member, representing the Anaconda Wire and Cable Co. and the National Electric Manufacturers Assn.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, J. H. Foote.
Vice-Chairman, W. R. Hibbard.
Secretary, H. H. Stout, Jr.

EMERGENCY ALTERNATE PROVISIONS AND EMERGENCY SPECIFICATIONS

The committee recommended to the Society, Emergency Alternate Provisions in the Tentative Specifications for Rope-Lay-Stranded and Bunch-Stranded Copper Cables for Electrical Conductors (B 158 – 41 T) which were accepted by Committee E-10 on Standards on November 28, 1941, and issued as a pink sticker under the designation EA – B 158. These changes were necessary to make more lenient the section on lay requirements of bunch-stranded conductors in order to conform with other existing standards.

During the year the committee also prepared and presented to the Society Emergency Specifications for Lead-Coated and Lead-Alloy-Coated Copper Wire for Electrical Purposes. These emergency specifications were accepted by Committee E-10 on March 27, 1942, and issued under the designation ES-1. Subsequent to this action, it was found necessary to revise certain sections of the specifications which had not been fully developed because of emergency conditions. A revision of the emergency specifications was approved under date of June 18, 1942.

During the past two years the committee has been preparing Specifications for Hard and Medium-Hard-Drawn Grades of Tinned Copper Wire to supplement the soft grade provided in the Standard Specifications for Tinned Soft or Annealed Copper Wire for Electrical Purposes (B 33 – 39). These specifications are now ready for consideration by the Society, but due to the non-availability of tin for general use and the probable lack of application for such specifications during the present emergency it seems to the committee wise to publish these specifications as information pending a more opportune time for their approval as a tentative standard. They are accordingly included in Appendix I to this report. Comments and suggestions on these proposed specifications are solicited by the committee.

RECOMMENDATIONS AFFECTING STANDARDS

The committee is submitting in this report three new tentative specifications which will replace one tentative standard, the revision and adoption as standard of a tentative standard, and the reaffirmation of the remaining standards and tentative standards under its jurisdiction with editorial revisions.

I. NEW TENTATIVE STANDARDS

Tentative Specifications for Rope-Lay-Stranded Copper Conductors Having Concentric-Stranded Members, for Electrical Conductors.[1]—The requirements covering rope-lay-stranded copper conductors having concentric-stranded members have been separated from the Tentative Specifications for Rope-Lay-Stranded and Bunch-Stranded Copper Cables (B 158 - 41 T) and incorporated in these new specifications which are recommended for publication as tentative.

Tentative Specifications for Rope-Lay-Stranded Copper Conductors Having Bunch-Stranded Members for Electrical Conductors.[1]—The requirements covering rope-lay-stranded copper conductors having bunch-stranded members have been separated from the Tentative Specifications B 158 - 41 T and incorporated in these new specifications which are recommended for publication as tentative.

Tentative Specifications for Bunch-Stranded Copper Conductors for Electrical Conductors.[1]—The requirements covering bunch-stranded copper conductors have also been separated from the Tentative Specifications B 158 - 41 T and incorporated in these new specifications which are recommended for publication as tentative.

II. ADOPTION OF TENTATIVE STANDARD AS STANDARD

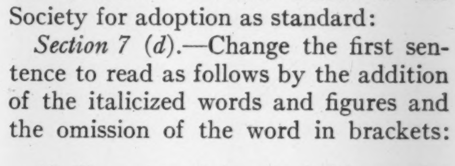
Tentative Specifications for Soft Rectangular and Square Copper Wire for Electrical Conductors (**B 48 - 40 T**).[2]—The committee recommends that these specifications, which have been published as tentative for three years, be revised as indicated below and approved for reference to letter ballot of the Society for adoption as standard:

Section 7 (*d*).—Change the first sentence to read as follows by the addition of the italicized words and figures and the omission of the word in brackets:

> (*d*) The corners of the wire shall be rounded to a quarter circle of the radius [as] *with a plus or minus tolerance of 25 per cent (see Fig. 1). The radius shall be* determined by a radius gage *as* prescribed in the following table:

Also in the tabulation at the end of the paragraph add a footnote reference "*b*" after the words "rounded edge" in columns 2 and 3 and add a new footnote to read as follows:

> [b] A rounded edge is an edge produced by rolling round wire to the size specified either with or without edging rolls (see Fig. 1).



NOTE.—The arc is not necessarily tangent to the flats at points *A*. However, the wire shall be commercially free of sharp, rough or projecting edges.

FIG. 1.—Corner of Wire Showing Rounded Edge.

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[1] These specifications were accepted as tentative by the Society and appear in the 1942 Book of A.S.T.M. Standards, Part I, pp. 1194, 1189, and 1199, respectively.

[2] 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 358.

III. WITHDRAWAL OF TENTATIVE STANDARD

The committee recommends that the Tentative Specifications for Rope-Lay-Stranded and Bunch-Stranded Copper Cables for Electrical Conductors (B 158 – 41 T)[3] be withdrawn since they have been superseded by the three proposed new tentative specifications mentioned earlier in this report.

IV. REAFFIRMATION OF STANDARDS

The committee has reviewed all the standards under its jurisdiction and recommends their continuation as standard with certain editorial changes referred to below in order to bring them in line with current practice:

Standard Specifications for Medium-Hard-Drawn Copper Wire (**B 2 – 40**),[4] *for Tinned Soft or Annealed Copper Wire for Electrical Purposes* (**B 33 – 39**),[5] *and for Hard-Drawn Copper Alloy Wires for Electrical Conductors* (**B 105 – 39**).[5]—Clarify and make consistent the number of decimal places and number of significant figures employed in the dimensional requirements in these specifications.

Standard Specifications for Hard-Drawn Copper Wire (**B 1 – 40**),[4] *for Medium-Hard-Drawn Copper Wire* (**B 2 – 40**),[4] *for Hard-Drawn Copper Alloy Wires for Electrical Conductors* (**B 105 – 39**),[5] *for Figure-9 Deep-Section Grooved and Figure-8 Copper Trolley Wire for Industrial Haulage* (**B 116 – 40**),[4] *for Copper Trolley Wire* (**B 47 – 39**),[5] *and for Bronze Trolley Wire* (**B 9 – 39**).[5]—Incorporate the following sentence in the notes relative to speed of testing appearing in these specifications:

> It is suggested that tests be made at speed of moving head which, under no-load conditions, are not greater than 3 in. per min., but in no case at a speed greater than that at which correct readings can be made.

Standard Specifications for Concentric-Lay-Stranded Copper Cable, Hard, Medium-Hard, or Soft (**B 8 – 41**).[6]—In these specifications change all references to "cable" to read "conductor."

Standard Specifications for Figure-9 Deep-Section Grooved and Figure-8 Copper Trolley Wire for Industrial Haulage (**B 116 – 40**).[4]—In Fig. 1 of these specifications change the word "die" maker to read "tool" maker.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 42 members; 40 members returned their ballots with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Item	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Spec. for Rope-Lay-Stranded Copper Conductors Having Concentric-Stranded Members, for Electrical Conductors	29	0	11
Spec. for Rope-Lay-Stranded Copper Conductors Having Bunch-Stranded Members for Electrical Conductors	29	0	11
Spec. for Bunch-Stranded Copper Conductors for Electrical Conductors	29	0	11
II. ADOPTION OF TENTATIVE STANDARD AS STANDARD			
Spec. for Soft Rectangular and Square Copper Wire for Electrical Conductors (B 48 – 40 T), as revised	31	0	9
III. WITHDRAWAL OF TENTATIVE STANDARD			
Spec. for Rope-Lay-Stranded and Bunch-Stranded Copper Cables for Electrical Conductors (B 158 – 41 T)	30	0	10

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[3] 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 369.

[4] 1940 Supplement to Book of A.S.T.M. Standards, Part I, pp. 150, 146, 154.

[5] 1939 Book of A.S.T.M. Standards, Part I, pp. 563, 568, 579, 573.

[6] 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 149.

This report has been submitted to letter ballot of the committee which consists of 42 members; 40 members returned their ballots, of whom 37 have voted affirmatively, and 0 negatively.

Respectfully submitted on behalf of the committee,

J. H. FOOTE,
Chairman.

G. E. DEAN,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee B-1 recommended to Committee E-10 on Standards that Emergency Provisions EA - B 158 be withdrawn inasmuch as the Tentative Specifications for Rope-Lay-Stranded and Bunch-Stranded Copper Cables for Electrical Conductors (B 158 - 41 T) to which they apply had been withdrawn by action at the annual meeting. This recommendation was accepted by Committee E-10 on August 24, 1942.

On December 11, 1942, Emergency Alternate Provisions in the Specifications for Concentric-Lay-Stranded Copper Conductors, Hard, Medium-Hard, and Soft (B 8 - 41), submitted by Committee B-1, were accepted by Committee E-10 and issued under the designation EA - B 8. They were published in ASTM BULLETIN, No. 120, January, 1943, p. 52, and have been issued in the form of a pink sticker for attachment to the standard.

APPENDIX

PROPOSED SPECIFICATIONS FOR TINNED HARD-DRAWN AND MEDIUM-HARD-DRAWN COPPER WIRE FOR ELECTRICAL PURPOSES[1]

These are proposed specifications and are published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia, Pa.

Scope

1. These specifications cover tinned hard-drawn and medium-hard-drawn round copper wire for electrical purposes.

Material

2. (*a*) *Tin for Coating.*—The coating material shall be commercially pure tin.

(*b*) *Copper Base Metal.*—The copper in the wire shall conform to the requirements of the standard specifications of the American Society for Testing Materials, as follows:

Standard Specifications for Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (A.S.T.M. Designation: B 5),[2] or

Standard Specifications for Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (A.S.T.M. Designation: B 4),[3] low resistance grade.

Or copper bars of special qualities, forms, or types, as may be agreed upon by the manufacturer and the purchaser, and which will conform to the requirements prescribed in these specifications.

Tensile Properties

3. (*a*) The finished or tinned wire shall conform to the requirements as to tensile properties prescribed in Table I. (Notes 1 and 2.)

(*b*) For wire the nominal diameter of which is more than 0.001 in. (1 mil) greater than a size listed in Table I, but which is less than that of the next larger size, the requirements of the next larger size shall apply.

(*c*) Tension tests shall be made on representative specimens. The elongation shall be determined as the increase in length at the time of break, due to breaking of the wire in tension, and shall be measured between the jaws of the testing machine (Note 3). The zero length shall be the distance between the jaws of the testing machine when a load equal to 10 per cent of the specified tensile strength has been applied and the final lengths shall be the distance between the jaws at the time of rupture. The zero length shall be as near 60 in. as practicable. The fracture shall be between the jaws of the testing machine and shall be not closer than 1 in. to either jaw.

(*d*) *Retests.*—If, upon testing a specimen from any coil or spool of wire, the results do not conform to the requirements prescribed in Table I, two additional specimens shall be tested, and the average of the three tests shall determine the acceptance or rejection of the coil or spool.

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[1] These proposed specifications are under the jurisdiction of the A.S.T.M. Committee B-1 on Copper and Copper-Alloy Wires for Electrical Conductors. Published as information, June, 1942.

[2] 1942 Book of A.S.T.M. Standards, Part I, p. 672.

[3] *Ibid.*, p. 676.

TABLE I.—TENSILE REQUIREMENTS OF COATED WIRE.[a]

Diameter, in.	Area at 20 C. (68 F.)		Weight, lb. per 1000 ft.	Tinned Hard-Drawn Wire		Tinned Medium-Hard-Drawn Wire		
	cir. mils	sq. in.		Tensile Strength, min., psi.	Elongation in 60 in., min., per cent	Tensile Strength, psi.		Elongation in 60 in., min., per cent
						min.	max.	
0.2043	41 740	0.03278	126.4	54 100	1.24	42 400	51 400	1.25
0.1819	33 100	0.02600	100.2	55 100	1.18	43 300	52 300	1.20
0.1620	26 250	0.02062	79.46	55 900	1.14	44 100	53 100	1.15
0.1443	20 820	0.01635	63.02	56 700	1.09	44 900	53 900	1.11
0.1285	16 510	0.01297	49.97	57 300	1.06	45 500	54 500	1.08
0.1144	13 090	0.01028	39.63	57 900	1.02	46 000	55 000	1.06
0.1019	10 380	0.008155	31.43	58 400	1.00	46 500	55 500	1.04
0.09074	8 234	0.006467	24.92	58 900	0.97	46 900	55 900	1.02
0.08081	6 530	0.005129	19.77	59 100	0.95	47 200	56 200	1.00
0.07196	5 178	0.004067	15.68	59 300	0.92	47 300	56 300	0.98
0.06408	4 107	0.003225	12.43	59 600	0.90	47 600	56 600	0.96
0.05707	3 257	0.002558	9.858	59 800	0.89	47 800	56 800	0.94
0.05082	2 583	0.002028	7.818	59 900	0.87	47 900	56 900	0.92
0.04526	2 048	0.001609	6.200	60 100	0.86	48 100	57 100	0.90
0.04030	1 624	0.001276	4.917	60 300	0.85	48 300	57 300	0.88
0.03589	1 288	0.001012	3.899	60 400	0.84	48 400	57 400	0.87
0.03196	1 022	0.0008023	3.092	60 600	0.83	48 600	57 600	0.86
0.02846	810.1	0.0006363	2.452	60 800	0.82	48 800	57 800	0.85
0.02535	642.4	0.0005046	1.945	61 000	0.81	49 000	58 000	0.84

[a] These values of tensile strength and elongation requirements for coated wire take account of changes occurring as the result of the application of coatings by hot-dipping methods. It is desirable that the physical values of coated wire conform as nearly as practical to the requirements prescribed in the Standard Specifications for Hard-Drawn Copper Wire (A.S.T.M. Designation: B 1)[4] or the Standard Specifications for Medium-Hard-Drawn Copper Wire (A.S.T.M. Designation: B 2)[5] of the American Society for Testing Materials.

Resistivity

4. Electrical resistivity shall be determined on representative specimens by resistance measurements (Note 4). At a temperature of 20 C. (68 F.) the resistivity of coated wire shall not exceed the values prescribed in Table II.

TABLE II.—RESISTIVITY REQUIREMENTS OF COATED WIRE.

Nominal Diameter, in.	Resistivity at 20 C. (68 F.), ohms (mile, pound)	
	Hard	Medium-Hard
0.204 to 0.103	943.92	938.85
0.102 to 0.025	951.72	946.06

Dimensions and Permissible Variations

5. (*a*) The size shall be expressed as the diameter of coated wire in decimal fractions of an inch; using three places of decimals, that is, in mils, for wire having a nominal diameter of 0.100 in. and larger; and not more than four places of decimals, that is, in tenths of mils, for wire having nominal diameters less than 0.100 in. (Note 2).

(*b*) The coated wire shall not vary from the specified diameter by more than plus 3 per cent or minus 1 per cent.

(*c*) The diameter of coated wire of each coil or spool shall be measured. In the case of coils, the diameter shall be measured at three places, one near each end and one near the middle of the coil. In the case of spools, approximately 12 ft. of the coated wire shall be unreeled and the diameter shall be measured at six places between the second and twelfth foot from one end. The average of the measurements obtained on any coil or spool shall be within the limits specified in Paragraph (*b*).

Continuity of Coating

6. The coating on the wire shall be continuous. The continuity of the tin coating shall be determined by the

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[4] 1942 Book of A.S.T.M. Standards, Part I, p. 686.

[5] *Ibid.*, p. 690.

sodium polysulfide - hydrochloric acid test as described in Section 7. (Note 5.)

Sodium Polysulfide - Hydrochloric Acid Test for Continuity of Coating

7. (*a*) *Length of Specimens.*—The test specimens shall each have a length of about 6 in. They shall be tagged or marked to correspond with the coil or spool from which they were cut.

(*b*) *Treatment of Specimens.*—The test specimens shall be thoroughly cleaned by immersion in benzine, redistilled petroleum ether, ether, or carbon tetrachloride for at least 3 min., then removed and wiped dry with a clean, soft cloth. The specimens thus cleaned shall be kept wrapped in a clean, dry cloth until required for test. That part of the specimen to be immersed in the test solution shall not be handled. Care shall be taken to avoid abrasion by the cut ends.

(*c*) *Special Solutions:*

Hydrochloric Acid Solution (*sp. gr. 1.088*).—Dilute HCl (sp. gr. 1.18) with distilled water to a specific gravity of 1.088 measured at 15.5 C. (60 F.). A portion of this HCl solution having a volume of 180 ml. shall be considered to be exhausted when the following number of test specimens of a length as indicated in Paragraph (*d*) have been immersed in it for two cycles:

Diameter, in.	Maximum Number of Specimens to be Tested for Two Cycles in 180 ml. of Acid Solution
0.2043 to 0.141	2
0.140 to 0.0851	4
0.085 to 0.0501	6
0.050 to 0.0381	10
0.038 to 0.0301	12
0.030 to 0.0254	14

Sodium Polysulfide Solution (*sp. gr. 1.142*).—Prepare a concentrated solution by dissolving sodium sulfide crystals in distilled water until the solution is saturated at about 21 C. (70 F.), adding about 250 g. of flowers of sulfur per liter, and allow to stand for at least 24 hr. Then dilute a portion of the concentrated solution with distilled water to a specific gravity of 1.142 at 15.5 C. (60 F.). The sodium polysulfide test solution should have sufficient strength to blacken thoroughly a piece of clean, untinned copper wire in 5 sec. A portion of the test solution used for testing specimens shall not be considered to be exhausted until it fails to blacken a piece of clean copper as described above.

(*d*) *Procedure.*—A length of at least 4½ in. from each of the cleaned specimens shall be immersed, in accordance with the following cycles, in test solutions maintained at a temperature between 15.5 and 21 C. (60 and 70 F.):

(*1*) The specimen shall be immersed for 1 min. in the HCl (sp. gr. 1.088), washed, and wiped dry.

(*2*) The specimen shall be immersed for 30 sec. in the sodium polysulfide (sp. gr. 1.142), washed, and wiped dry.

(*3*) The specimen shall be immersed for 1 min. in the HCl (sp. gr. 1.088), washed, and wiped dry.

(*4*) The specimen shall be immersed for 30 sec. in the sodium polysulfide (sp. gr. 1.142), washed, and wiped dry.

Washing Specimens.—After each immersion, the specimens shall immediately be thoroughly washed in clean water and wiped dry with a clean soft cloth.

(*e*) *Examination of Specimens.*—After the operations described in Paragraph (*d*), the specimens shall be examined to ascertain if copper exposed through openings in the tin coating has been blackened by action of the sodium polysulfide. The specimens shall be considered to have failed if by such blackening exposed copper is revealed. No attention shall be paid to blackening within 0.5 in. of the cut end.

Mechanical Test for Adhesion of Coating

8. A mechanical test for adhesion of tinning may be made by bending the wire around a rod having a diameter equal to four times the diameter of the wire, and dipping the bent portion of the wire only in the sodium polysulfide solution (Section 7 (*c*)), for 30 sec. Any cracking or parting of the coating shown by blackening of the copper shall be cause for rejection.

Joints

9. Joints shall not be made in the wire after it has been drawn to size or coated. Joints in the wire or rods prior to final drawing shall be made in accordance with the best commercial practice. (Note 6.)

Finish

10. (*a*) The tin coating shall consist of a smooth, continuous layer, firmly adherent to the surface of the copper.

(*b*) The wire shall be free of all imperfections not consistent with the best commercial practice.

Packing and Shipping

11. (*a*) Package sizes shall be agreed upon by the manufacturer and the purchaser in the placing of individual orders (Note 7).

(*b*) The coated wire shall be protected against damage in ordinary handling and shipping.

Inspection

12. All tests and inspection shall be made at the place of manufacture, unless otherwise especially agreed upon by the manufacturer and the purchaser at the time of purchase. The manufacturer shall afford the inspector representing the purchaser all reasonable facilities, without charge, to satisfy him that the material is being furnished in accordance with these specifications.

Rejection

13. Wire that fails to conform to the requirements of these specifications shall be rejected.

EXPLANATORY NOTES

NOTE 1.—The values of coated wire diameter in Table I, which correspond to gage numbers of the American wire gage, are given in four significant figures for reference and in the interest of greater precision in making calculations of weight, area, and resistivity. In specifying diameters of coated wire, or in inspecting wire, the diameter should be expressed in not more than three, nor less than two, significant figures within the range of sizes included in Table I and in accordance with the requirements of Section 5. The use of gage numbers to specify wire sizes is not recognized in these specifications because of the possibility of confusion. An excellent discussion of wire gages and related subjects is contained in *Circular No. 31* of the National Bureau of Standards.[6]

NOTE 2.—Other tests than those provided in these specifications have been considered at various times, such as twist tests, wrap tests, etc. It is the opinion of the committee that twist tests on hard-drawn or medium-hard-drawn wire serve no useful purpose and that wrap tests, other than that provided in Section 8 which is a test for adhesion, are likewise undesirable and inconclusive as to results and significance.

NOTE 3.—It is known that the rate of loading during tension testing of copper wire affects the performance of the specimen to a greater or lesser extent, depending upon many factors. In general, values of tensile strength as determined by test are increased and values of elongation are reduced with increase of speed of the moving head of the testing machine. These effects are pronounced when the speed of the moving head is excessive in the testing of hard-drawn and medium-hard-drawn wires. It is suggested that tests on such wire be made at speeds of moving head which, under no-load conditions, are not greater than 3 in. per min., but in no case at a greater speed than that at which correct readings can be made.

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[6] *Circular No. 31*, Nat. Bureau Standards, is for sale by the Superintendent of Documents, Government Printing Office, Washington, D. C., price 20 cents.

NOTE 4.—"Resistivity" is used in place of "conductivity." The value of 0.15328 ohm (meter, gram) at 20 C. (68 F.) is the international standard for the resistivity of annealed copper, equal to 100 per cent conductivity. This term means that a wire 1 m. in length and weighing 1 g. would have a resistance of 0.15328 ohm. This is equivalent to a resistivity value of 875.20 ohms (mile, pound) which signifies the resistance of a wire 1 mile in length weighing 1 lb. It is also equivalent, for example, to 1.7241 microhms per centimeter of length of a bar, 1 sq. cm. in cross-section. A complete discussion of resistivity is contained in *Circular No. 31* of the National Bureau of Standards.[6] Because the specific resistance of tin is greater than copper and because the relative amount of tin is greater on fine wire than on wire of larger diameter, the resistivity of tin-coated copper wire varies inversely with the diameter. Relationships that may be useful in connection with the values of resistivity prescribed in these specifications are as given in Table III, each column containing equivalent expressions, at 20 C. (68 F.).

TABLE III.—RESISTIVITY VALUES.

Conductivity at 20 C. (68 F.) per cent	100.00	93.22	92.72	92.51	91.96
Ohms (mile, pound)	875.20	938.85	943.92	946.06	951.72
Ohm (meter, gram)	0.15328	0.16443	0.16532	0.16569	0.16668
Ohms (mil, foot)	10.371	11.125	11.185	11.211	11.278
Ohm (meter, square millimeter)	0.017241	0.018495	0.018595	0.018637	0.018748
Microhm—inch	0.67879	0.72816	0.73209	0.73375	0.73814
Microhms—centimeter	1.7241	1.84949	1.85947	1.86369	1.87484

NOTE 5.—The continuity of the pure tin coating on copper wire and not the thickness of the coating is essential for the protection of the wire against corrosion of certain insulations. Although the thickness of the tin coating on the same wire varies, the thickness on the various sizes of wire applied under similar conditions, except the very fine sizes, is approximately the same. The number of cycles of the continuity test should not vary with the diameter of the wire. As a precaution to detect partially coated areas, two cycles of the test are specified.

NOTE 6.—Mechanical joints made during inspection at the request of the purchaser are permissible, if agreed upon at the time of placing the order.

NOTE 7.—The manufacturer and the purchaser should agree on package sizes which will be sufficiently large and yet not so heavy or bulky that the wire may likely be damaged in handling.

REPORT OF COMMITTEE B-2
ON
NON-FERROUS METALS AND ALLOYS

Committee B-2 on Non-Ferrous Metals and Alloys held one meeting, on June 24, 1941, in Chicago, Ill., during the 1941 annual meeting of the Society. The Advisory Committee held two meetings, one on the above-mentioned date, and the other in New York, N. Y., on February 11, 1942. The Advisory Committee approved numerous changes in membership, discussed matters before the various subcommittees, and made the following recommendations to the Joint Coordinating Committee of Committees A-1, A-10, B-2, and B-4 which were later approved: (*a*) That specifications for contact metals, on account of their principal use, should be prepared in Committee B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys rather than by Subcommittee V on Precious Metals and Alloys of Committee B-2; (*b*) That specifications on nickel-chromium-iron alloys (such as the several Tentative Specifications for Nickel-Chromium-Iron Alloy B 166, B 167, and B 168), wherein iron is a minor constituent, are within the proper scope of Subcommittee VII on Refined Nickel and High Nickel Alloys of Committee B-2 rather than of Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys, whose scope, as stated, is "iron-base alloys termed 'stainless' or 'rustless'."

The committee also considered the proposal that the Standard Specifications for Slab Zinc (B 6 – 37) and for Rolled Zinc (B 69 – 39) be submitted to the American Standards Association for approval as American standard. The chairman of Subcommittee IV on Refined Zinc and Wrought Zinc, E. H. Bunce, reported that he had discussed the matter with several technical men in the zinc industry and he concurred in their general feeling that no revision of these standards is contemplated and that they could properly be recommended to the A.S.A.

Subsequent to the 1941 annual meeting, Committee B-2 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative Specifications for:

Nickel Rods and Bars (B 160 – 41 T),
Nickel Cold-Drawn Pipe and Tubing (B 161 – 41 T),
Nickel Plate, Sheet, and Strip (B 162 – 41 T),
Nickel, Nickel-Copper Alloy, and Nickel-Chromium-Iron Alloy Seamless Condenser Tubes and Ferrule Stock (B 163 – 41 T),
Nickel-Copper Alloy Rods and Bars (B 164 – 41 T),
Nickel-Copper Alloy Cold-Drawn Pipe and Tubing (B 165 – 41 T),
Nickel-Chromium-Iron Alloy Rods and Bars (B 166 – 41 T),
Nickel-Chromium-Iron Alloy Cold-Drawn Pipe and Tubing (B 167 – 41 T),
Nickel-Chromium-Iron Alloy Plate, Sheet, and Strip (B 168 – 41 T),

Revision of Tentative Specifications for:

Nickel-Copper Alloy Plate, Sheet, and Strip (B 127 – 39 T), and
Electrolytic Cathode Copper (B 115 – 38 T).

These recommendations were accepted[1] by Committee E-10 on August 25, 1941, and the new tentative specifications were assigned the A.S.T.M. serial designations indicated above. The new and revised tentative standards appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part I.

Committee B-2 also submitted to Committee E-10 the following three additional recommendations which were accepted[1] on January 17, 1942:

New Tentative Specifications for:

Oxygen-Free Electrolytic Copper Wire Bars, Billets, and Cakes,

Tentative Revision of Standard Specifications for:

Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (B 4 – 27), and

Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (B 5 – 27).

The new tentative specifications have been issued as a separate reprint bearing the designation B 170 – 42 T. The tentative revisions of Standard Specifications B 4 and B 5 appeared in the ASTM BULLETIN No. 114, January, 1942, p. 35.

I. ADOPTION OF TENTATIVE STANDARD AS STANDARD

The committee recommends, as proposed by Subcommittee VIII on Miscellaneous Refined Metals and Alloys, that the Tentative Specifications for Phosphor Tin (B 51 – 38 T)[2] be approved, without revision, for reference to letter ballot of the Society for adoption as standard.

II. ADOPTION AS STANDARD OF TENTATIVE REVISIONS OF STANDARDS

The committee recommends that the tentative revisions[3] of the Standard Specifications for Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (B 4 – 27) and for Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (B 5 – 27) be approved without change for reference to letter ballot of the Society for adoption as standard.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 115 members; 73 members returned their ballots with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. ADOPTION OF TENTATIVE STANDARD AS STANDARD			
Spec. for Phosphor Tin (B 51 – 38 T)	45	0	28
II. ADOPTION AS STANDARD OF TENTATIVE REVISIONS OF STANDARDS			
Spec. for Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (B 4 – 27)	57	0	16
Spec. for Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (B 5 – 27)	57	0	16

EMERGENCY STANDARD

Emergency Specifications for Fire-Refined Copper for Wrought Products and Alloys prepared by the committee were accepted by Committee E-10 on Standards on June 13, 1942, under the emergency procedure, and published under the designation ES-7.[4]

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee B-2 reported the following results of the letter ballot vote of a total of 97 ballots returned from a committee membership of 117: B 160, affirmative 47, negative 0, ballots marked "not voting" 50; B 161 and B 162, affirmative 46, negative 0, ballots marked "not voting" 51; B 163, affirmative 49, negative 0, ballots marked "not voting" 48; B 164, affirmative 48, negative 0, ballots marked "not voting" 49; B 165, affirmative 46, negative 0, ballots marked "not voting" 51; B 166, affirmative 47, negative 0, ballots marked "not voting" 50; B 167, affirmative 45, negative 0, ballots marked "not voting" 52; B 168, affirmative 46, negative 0,ballots marked "not voting" 51; B 127, affirmative 47, negative 0, ballots marked "not voting" 50; B 115, 62 ballots returned, affirmative 45, negative 0, ballots marked "not voting" 17; B 170, 80 ballots returned, affirmative 58, negative 1, ballots marked "not voting" 21; B 4 and B 5, 72 ballots returned, affirmative 50, negative 0, ballots marked "not voting" 22.

[2] 1939 Book of A.S.T.M. Standards, Part I, p. 1143.

[3] Published in ASTM BULLETIN, No. 114, January, 1942, p. 35

[4] These emergency specifications appear in the 1942 Book of A.S.T.M. Standards, Part I, p 957.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Refined Copper (J. L. Christie, chairman) on June 24, 1941, reconsidered the action referred to in the 1941 Report recommending that the Tentative Specifications for Electrolytic Cathode Copper (B 115 – 38 T) be approved for adoption as standard, and in place of that recommendation it decided to continue the specifications as tentative with a revision to lower the maximum allowed resistivity from 0.15436 international ohms per meter-gram to a new value of 0.15328 international ohms per meter-gram. This corresponds to an increase in the minimum permissible conductivity from 99.3 per cent to 100 per cent. Also an editorial change was made in Section 6 (*b*) on sampling to require that scale and surface dirt shall be rejected.

After favorable letter ballot by Committee B-2, these changes were accepted by Committee E-10 on August 25, 1941, as mentioned earlier in this report.

Corresponding changes in the resistivity values in Standard B 4 – 27 for lake copper wire bars and in Standard B 5 – 27 for electrolytic wire bars were recommended by the subcommittee for publication as tentative. This recommendation was approved by letter ballot in Committee B-2 and the proposed tentative revisions were accepted by Committee E-10 on January 17, 1942.

New Tentative Specifications for Oxygen-Free Electrolytic Copper Wire Bars, Billets, and Cakes, prepared by a special subgroup under the leadership of Carter S. Cole, were accepted by Committee E-10 on January 17, 1942, and issued under the designation B 170 – 42 T.

Information was received that a high grade of fire-refined copper trade-named "Braden," formerly shipped from South America to England, is now coming into the United States, and is acceptable to the Ordnance Department for use in cartridge brass and gilding metal (bullet jackets). It appeared that emergency alternate provisions in the Standard Specifications for Fire-Refined Copper Other than Lake (B 72 – 33), or possibly emergency specifications for this grade of copper would be desirable during the period of National Emergency, particularly since it was expected that similar copper would shortly be available from other sources. A small group was accordingly appointed to formulate the proposed requirements, but difficulty was encountered in defining the desired requirements of material that would be acceptable both to the producer and the U. S. Ordnance Department. Agreement was eventually secured and the specifications have now been approved by the following letter ballot vote of the subcommittee: From a membership of 34, 25 members returned their ballots, 24 of whom have voted affirmatively, 0 negatively, and 1 member marked his ballot "not voting." Upon approval by E. E. Thum, as chairman of Committee B-2, these specifications have been referred to Committee E-10 on Standards for approval under the procedure for emergency provisions and standards.

Subcommittee III on White Metals and Alloys (G. H. Clamer, chairman) at a meeting in Chicago on June 24, 1941, gave consideration to the table of impurities in Section 4 of the Tentative Specifications for Soft Solder Metal (B 32 – 40 T). It was agreed that the stated allowances for iron, zinc, and aluminum in grade A and grade B be revised as soon as satisfactory methods for the determination of minute amounts can be approved by Committee E-3 on Methods of Chemical Analysis of Metals. (Messrs. Hiers and Behr of the National Lead Co. reported that their laboratory has developed methods having a degree of accuracy making possible the determination of aluminum and zinc content of 0.0003 per cent if a 10-g. sample is used.) It was agreed that paragraph (*b*) under "Scope" shall remain, pending the development of the above referred to analytical methods. It was also agreed and accepted by Committee B-2 that the word "none" shall apply to zinc and aluminum in grade A for both tin-lead and tin-lead-antimony solders; the word "none" to be followed by a mark referring to a footnote as follows: "The term 'none' as applied to aluminum and zinc allowances is defined as 0.0003 per cent when determined on a 10-g. sample."

At meetings held in Cleveland on March 4 and in New York on April 1, 1942, emergency alternate provisions in Specifications B 32 which show the typical tin-lead alloy compositions (and their melting ranges) containing less than the 30 per cent tin permissible by governmental regulations, and include in addition eight commercial alloys of tin-lead-bismuth or tin-lead-silver which conform to these limits were approved. These were accepted by the Society on April 6, 1942, and published in the form of a pink sticker (EA - B 32) for attachment to Tentative Specifications B 32 - 40 T.

A footnote in the emergency alternate provisions indicates that all these alternate solders are hand or dipping solders and that none can be used for making wiped joints. However, very recently, it has been found that the following two alloys have been used with some success as wiping solders:

	Alloy A	Alloy B
Tin, per cent	30	20
Lead, per cent	68	65
Bismuth, per cent	..	15
Antimony, per cent	1.8	..
Arsenic, max., per cent	0.1	..

The Standard Specifications for White Metal Bearing Alloys (Known Commercially as "Babbitt Metal") (B 23 - 26) have been under discussion for over a year. At the June 24, 1941, meeting, agreement was reached in principle, but specific consideration of what would be desirable for a peace-time economy has been complicated by the urgent requirements to economize on the strategic metal tin, whereby a 12 per cent limitation is obligatory, thus making alloy grades 1 to 6 inclusive of Standard B 23 - 26 unprocurable except by special allocation. At a meeting held in Cleveland on March 4, 1942, it was decided that data concerning the chemical composition and reported physical properties of several commercial "babbitts" low in tin be collected and issued as Emergency Alternate Provisions. These were accepted by the Society on April 6, 1942, and published in the form of a pink sticker (EA - B 23) for attachment to Standard Specifications B 23 - 26.

Subcommittee VII on Refined Nickel and High Nickel Alloys, Cast and Wrought has elected O. B. J. Fraser permanent chairman. Nine new specifications prepared by this subcommittee during the previous year were accepted by Committee E-10 on August 25, 1941. The titles and designations assigned to these specifications are given earlier in this report.

Revisions of the Tentative Specifications for Nickel-Copper Alloy Plate, Sheet and Strip (B 127 - 39 T), noted in last year's report, were also accepted by Committee E-10.

Subcommittee VIII on Miscellaneous Refined Metals and Alloys (E. E. Schumacher, chairman) has recommended that the Tentative Specifications for Phosphor Tin (B 51 - 38 T) be adopted as standard.

The present officers of the committee were re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 115 members; 73 members returned their ballots, of whom 68 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

E. E. THUM,
Chairman.

C. E. SWARTZ,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee B-2 recommended changes in the Emergency Alternate Provisions in Standard Specifications for Babbitt Metal (EA – B 23) and in Tentative Specifications for Soft Solder Metal (EA – B 32), referred to in this report, which were accepted by Committee E-10 on Standards on January 5, 1943. The revised emergency provisions were published in ASTM BULLETIN, No. 120, January, 1943, p. 53, under the designations EA – B 23a and EA – B 32a, and have been issued in the form of pink stickers for attachment to the specifications to which they apply.

REPORT OF COMMITTEE B-3

ON

CORROSION OF NON-FERROUS METALS AND ALLOYS

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys held a meeting in Chicago, Ill., on June 24, 1941.

The committee consists of 74 members, of whom 62 are voting members; 28 are classified as producers, 24 as consumers, and 10 as general interest members.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Total Immersion Test (A. W. Tracy, chairman).—A statistical analysis of the data obtained by the cooperating laboratories in the total immersion tests on one metal—copper—in normal solutions of sulfuric acid, sodium chloride, and sodium hydroxide, aerated and nonaerated, indicated that agreement between individual test results at any one laboratory was good, but from one laboratory to another the test results were not under statistical control. Further work must be done before a proposed standard method of test can be recommended.

Subcommittee II on Alternate Immersion Test (C. M. Sterne, chairman).—The chairman has inspected the equipment for alternate immersion testing used at several member laboratories but due to pressure of war work no program has been formulated.

Subcommittee III on Spray Test (E. H. Dix, Jr., chairman).—Since the Tentative Method of Salt Spray Testing of Non-Ferrous Metals (B 117 - 41 T)[1] was revised last year, several additional suggestions regarding this tentative method have been received, indicating that another revision should be considered in the near future. Mr. C. E. Heussner will take over the chairmanship of subcommittee III this year as arranged at the March, 1941, meeting.

Subcommittee V on Statistical Analysis and Planning of Corrosion Testing (W. E. Campbell, chairman).—This subcommittee held a meeting in Washington, D. C., on February 4, 1942. It has supervised the calculation of statistical constants for data from the 10-yr. atmospheric corrosion tests, being carried on by Subcommittee VI, and will cooperate further in the calculation of data from tension test results of specimens cut from the 9 by 12-in. plates.

Statistical control charts of the total immersion test data from Subcommittee I were prepared and discussed at the February meeting.

The subcommittee aided in the planning of galvanic couple corrosion tests being carried out by Subcommittee VIII.

Subcommittee VI on Atmospheric Corrosion (W. H. Finkeldey, chairman).—This subcommittee has under way a series of atmospheric corrosion tests on 24 non-ferrous metals and alloys[2] which have been exposed since 1931 to various atmospheres at nine test locations.[3]

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[1] 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 566.

[2] A full description of the metals and alloys included in these tests, giving chemical composition, mechanical properties and microstructure, appears in the 1932 Report of Committee B-3, *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part I, p. 214 (1932).

[3] The results of previous tests, made after 1-yr., 3-yr., and 6-yr. exposures, are given in the 1933, 1935, and 1938 Reports of Committee B-3, *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 234 (1933); Vol. 35, Part I, p. 142 (1935); Vol. 38, Part I, p. 194 (1938).

The subcommittee has removed a group of specimens from these test racks after an exposure of ten years. Tension tests have been completed on the tension specimens and a statistical analysis of the results made. The 9 by 12-in. plates have been weighed by the National Bureau of Standards in accordance with the procedure followed after previous removals.

The subcommittee held a meeting in Washington on February 4, 1942, to make a visual inspection of the 9 by 12-in. plates. The written reports of the various observers covering this inspection are now being edited by H. S. Rawdon. Kodachrome transparencies have been made through the courtesy of the Eastman Kodak Co. of a selection of 9 by 12-in. plates of the various metals to obtain a visual record of typical surface conditions.

It is planned to remove corrosion products from the 9 by 12-in. plate specimens and reweigh them. The plates will then be cut up, formed into tension specimens, and tension tests made on them in a manner similar to the tests made on specimens exposed to the atmosphere as tension specimens. A statistical analysis of these test results will be made as soon as the complete data are available.

The work of the special subgroup appointed to survey the original plans for statistical analysis of the atmospheric corrosion data of Subcommittee VI has been continued in Subcommittee V. It is hoped that this work will have progressed far enough on the completion of the work associated with the removal of the 10-yr. specimens to permit the submission of a formal report.

Subcommittee VII on Weather (F. L. LaQue, chairman). This subcommittee held an organization meeting on February 4, 1942, in Washington, D. C., at which it was decided to draft a program looking towards a more precise evaluation of those weather factors which influence atmospheric corrosion and a correlation of such factors with the performance of materials under test. The ultimate aim would be to extend the basis for the intelligent application of results of atmospheric corrosion tests, particularly in respect to their use in estimating performance of materials at locations other than those at which large scale tests are made.

The activities of this subcommittee will be confined in the immediate future to the working out of the details of a program which can be put into effect after the war.

Mr. E. A. Anderson will serve as vice-chairman and Mr. C. H. Sample as secretary of this subcommittee.

Subcommittee VIII on Galvanic and Electrolytic Corrosion (L. J. Gorman, chairman).—The stainless steel specimens coupled with other metals for galvanic corrosion have been exposed to the atmosphere at four test locations, as follows:

	Exposure Date
Wilmington, N. C.	June 5, 1941
New York, N. Y.	June 20, 1941
State College, Pa.	December 5, 1941
Altoona, Pa.	December 6, 1941

The exposure of specimens at the La Jolla, Calif., test site has been held up pending the repair of test racks.

The stainless steels on test and the metals with which they are coupled are:

Stainless steel, type 304, 18 per cent chromium, 8 per cent nickel
Stainless steel, type 316, 18 per cent chromium, 8 per cent nickel, 2 to 3 per cent molybdenum
Aluminum 2 S ½ H
Aluminum alloy 24S-T
Aluminum alloy 53S-T
Copper, electrolytic, tough pitch
Mild steel
Lead, 6 per cent antimony
Zinc, prime western
Monel metal
Architectural bronze

Committee B-3 by letter ballot approved the organization of an Advisory Committee on Corrosion to advise with and to coordinate the work of the several A.S.T.M. committees dealing with corrosion of metals.

The present officers of the committee have been re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 62 voting members; 49 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

SAM TOUR,
Chairman.

A. W. TRACY,
Secretary

REPORT OF COMMITTEE B-4
ON
ELECTRICAL-HEATING, ELECTRICAL-RESISTANCE AND ELECTRIC-FURNACE ALLOYS

Committee B-4 on Electrical-Heating, Electrical-Resistance, and Electric-Furnace Alloys held three meetings during the year: in Chicago, Ill., on June 23, 1941, in Washington, D.C., on September 25 and 26, 1941, and in New York, N. Y., on February 12 and 13, 1942.

The committee is cooperating with the Joint A.S.M.E.-A.S.T.M. Research Committee on Effect of Temperature on the Properties of Metals, the Alloy Casting Institute, and Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys in the study of the behavior of metals at high temperatures and in the preparation of methods of testing.

The committee consists of 56 members, of whom 28 are classified as producers, 23 as consumers, and 5 as general interests.

The work of Section B of Subcommittee II on Contact Materials has increased so rapidly that the section has been organized as a new Subcommittee X, with F. E. Carter as chairman and S. G. Eskin as secretary.

A new Subcommittee VI on Editorial Review has been appointed, with H. L. Curtis as chairman and H. T. Wensel.

In Subcommittee V on Wrought and Cast Alloys for High-Temperature Use a new Section on Specifications has been appointed, with P. H. Brace as chairman, to prepare specifications for alloy bars and castings for high-temperature use.

On the new A.S.T.M. Advisory Committee on Corrosion, Dean Harvey has been appointed the official representative of Committee B-4, with L. L. Wyman as alternate.

The compilation of A.S.T.M. Standards on Electrical Heating and Resistance Alloys prepared by Committee B-4 has been in considerable demand, and most of the first edition of 750 copies, issued in September, 1941, has been disposed of. A new edition will be issued this year.

I. NEW TENTATIVE STANDARDS

The committee recommends that the new proposed Specifications for Round Nickel Wire for Lamps and Electronic Devices[1] be accepted for publication as tentative. These specifications are needed to specify the quality of the wire used for supports for filaments in lamps and electronic tubes, and grid rods in tubes.

II. REVISIONS OF TENTATIVE STANDARDS

The committee recommends that the following tentative methods be revised as appended hereto[2] and continued as tentative:

Tentative Methods of Testing:

Nickel and Nickel-Alloy Wire and Ribbon for Electronic Tube Filaments (B 118 - 39 T), and

Lateral Wire for Grids of Electronic Devices (B 156 - 41 T).

III. ADOPTION OF TENTATIVE STANDARD AS STANDARD

The committee recommends that Tentative Methods of Testing Sleeves and Tubing for Radio Tube Cathodes (B 128 - 40 T)[3] be approved for reference to letter ballot of the Society for adoption as standard.

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[1] These specifications were accepted as tentative by the Society and appear in the 1942 Book of A.S.T.M. Standards, Part I, p. 1433.

[2] These revised methods were accepted as tentative by the Society and appear in their latest revised form in the 1942 Book of A.S.T.M. Standards, Part I, pp. 1436 and 1442, respectively.

[3] 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 448.

IV. REAFFIRMATION OF STANDARDS

The following methods of test have been reviewed and the committee recommends that they be continued as standard without revision:

Standard Methods of Test for:[4]

Resistivity of Metallic Materials (B 63 – 36),
Thermoelectric Power of Electrical-Resistance Alloys (B 77 – 33), and
Temperature-Resistance Constants of Alloy Wires for Precision Resistors (B 84 – 36).

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 56 members, 45 members returned their ballots, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARD			
Spec. for Round Nickel Wire for Lamps and Electronic Devices	38	0	7
II. REVISION OF TENTATIVE STANDARDS			
Test for Nickel and Nickel-Alloy Wire and Ribbon for Electronic Tube Filaments (B 118 – 39 T)	39	0	6
Test for Lateral Wire for Grids of Electronic Devices (B 156 – 41 T)	39	0	6
III. ADOPTION OF TENTATIVE STANDARD AS STANDARD			
Test for Sleeves and Tubing for Radio Tube Cathodes (B 128 – 40 T)	39	0	6
IV. REAFFIRMATION OF STANDARDS			
Test for Resistivity of Metallic Materials (B 63 – 36)	42	0	3
Test for Thermoelectric Power of Electrical Resistance Alloys (B 77 – 33)	42	0	3
Test for Temperature-Resistance Constants of Alloy Wires for Precision Resistors (B 84 – 36)	42	0	3

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Life Tests (J. W. Harsch, chairman. — Interlaboratory check life tests on heater wire are being made in four laboratories, to check their equipment and test procedure. The National Bureau of Standards has set up a life test board to make standard tests. They also will carry in stock for sale, a small quantity of the 80-per cent nickel, 20-per cent chromium wire of known life for use as a comparison standard.

Subcommittee II on Electrical Properties (P. H. Dike, chairman).—This subcommittee has reviewed the Standard Tests for Resistivity of Metallic Materials (B 63 – 36), for Thermoelectric Power of Electrical-Resistance Alloys (B 77 – 33), and for Temperature-Resistance Constants of Alloy Wires for Precision Resistors (B 84 – 36)[4] and has found that no changes are needed in these standards at present.

Subcommittee V on Wrought and Cast Alloys for High Temperature Use (J. W. Harsch, chairman).—In connection with the study being made by the Office of Production Management (now the War Production Board) of the percentage of nickel needed in heat-resisting alloys for the principal high-temperature applications, Committee B-4 submitted a report with recommendations in July, 1941. This report was used in the preparation of recommendations submitted to the W. P. B. in March, 1942, by a meeting called by the Board, attended by furnace manufacturers, alloy-casting manufacturers, and petroleum producers, at which a number of members of the committee were present.

The subcommittee has continued the study of the significant properties and methods of test of alloys for use at high temperatures (over 1000 F.). Preliminary work has been done in the preparation of specifications for alloy castings of the 24 per cent chromium, 12 per cent nickel, balance iron type for structural elements, containers, and supports in electric furnaces. This work is being carried on in cooperation with the Alloy Casting Institute, the Joint Committee on the Effect of Temperature on the

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[4] 1939 Book of A.S.T.M. Standards, Part I, pp. 713, 726, 716.

Properties of Metals, and A.S.T.M. Committee A-10.

Proposed Specifications for Chromium-Nickel-Iron Alloy Castings for High-Temperature Service are being published in Appendix I as information for comment. Recent investigations have indicated that a further study of the magnetic permeability test is necessary to determine its scope and limitations as a criterion primarily of creep strength at high temperature. This study is being actively carried on.

Subcommittee VII on Thermostat Metals (P. H. Brace, chairman).—The study of methods of determining elastic properties of thermostat metals has been continued. Data are being obtained on the bend test as an indication of the temper of thermostat metal.

Subcommittee VIII on Metallic Materials for Radio Tubes and Incandescent Lamps (A. L. Chapman, chairman).—The new proposed Tentative Specifications for Round Nickel Wire for Lamps and Electronic Devices[1] mentioned earlier in this report have been prepared by this subcommittee. The Tentative Methods of Testing Nickel and Nickel-Alloy Wire and Ribbon for Electronic Tube Filaments (B 118 - 39 T) and Lateral Wire for Grids of Electronic Devices (B 156 - 41 T) have been revised in minor details[2] in order to remove certain differences between them, and the revised methods are being continued as tentative as proposed earlier in this report. This subcommittee has also recommended the adoption as standard of the Tentative Methods of Testing Sleeves and Tubing for Radio Tube Cathodes (B 128 - 40 T).[3]

Subcommittee IX on Methods of Test for Alloys in Controlled Atmospheres (P. H. Brace, chairman).—The test work on penetrating corrosion of resistor alloys in combusted gas atmospheres has been continued. Proposed Method of Test for Effect of Controlled Atmospheres Upon Alloys in Electric Furnaces is included as information in Appendix II.

At the request of Subcommittee V, work is being undertaken on the corrosion testing of castings to determine the effect of various controlled atmospheres upon various grades of heat-resisting alloys.

Subcommittee X on Contact Materials (F. E. Carter, chairman).—The new life test machine for testing contact materials has been purchased by twelve laboratories and has been in use since December, 1941. Minor details of construction have been improved and calibration tests have been made. The machine is giving excellent results. A program of testing has been prepared for four groups, each consisting of three laboratories, comprising life tests with different values of closing force and opening force, and test for load-carrying capacity. Tests also will be made to determine the effect of copper addition to the silver upon the life and load-carrying capacity.

A study is being made of the large number of sizes of solid contact rivets with a view to the preparation of a much smaller list of standard sizes. There appears in Appendix III a progress report on the standardization of contact rivets.

An abstract bibliography of articles on contact materials is also being prepared.

The present officers were re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 56 members; 45 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

DEAN HARVEY,
Chairman.

F. E. BASH,
Secretary.

APPENDIX I

PROPOSED SPECIFICATIONS FOR CHROMIUM-NICKEL-IRON ALLOY CASTINGS FOR HIGH-TEMPERATURE SERVICE[1]

These are proposed specifications and are published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia, Pa.

Scope

1. These specifications cover iron-base, heat-resisting, alloy castings of the 24 per cent chromium, 12 per cent nickel type, for temperatures up to 2000 F. These castings are intended for structural elements, containers, and supports in electric furnaces and similar applications.

Process

2. The castings shall be made by the electric-furnace process.

Heat Treatment

3. When specified, the castings shall be heat-treated as agreed upon by the manufacturer and the purchaser.

Chemical Composition

4. The castings shall conform to the following requirements as to chemical composition:

Nickel,[a] per cent	10.00 to 14.00
Chromium, per cent	23.00 to 28.00
Carbon, per cent	0.20 to 0.45
Nitrogen, max., per cent	0.20
Manganese, max., per cent	1.75
Phosphorus, max., per cent	0.05
Sulfur, max., per cent	0.05
Silicon, max., per cent	1.75
Total other elements, except iron, max., per cent	0.50

[a] Commercial nickel usually carries a small amount of cobalt, and within the usual limits, cobalt shall be counted as nickel.

Ladle Analysis

5. An analysis of each melt of alloy shall be made by the manufacturer to determine the percentages of nickel, chromium, carbon, nitrogen, manganese, and silicon. This analysis shall be made on drillings taken not less than ¼ in. beneath the surface of a test ingot made during the pouring of the melt. The chemical composition thus determined shall be reported to the purchaser or his representative, and shall conform to the requirements specified in Section 4.

Check Analysis

6. An analysis may be made by the purchaser from drillings taken from a broken tension test specimen or from a casting representing each melt. Drillings for analysis shall be taken not less than ¼ in. beneath the surface, except in the case of castings which are too thin. The chemical composition thus determined shall conform to the requirements specified in Section 4.

Tensile Properties After Aging

7. (*a*) The specimens shall be heated for 24 hr. at 1400 F. and shall be cooled in the furnace to a temperature below 400 F. They shall then be tested at room temperature, and shall conform to the following requirements:

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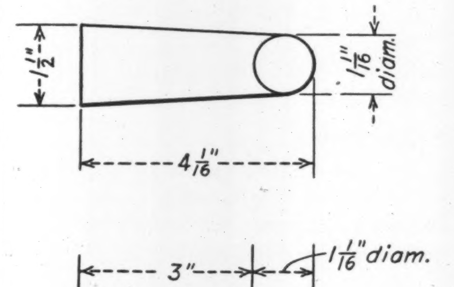
[1] These proposed specifications are under the jurisdiction of the A.S.T.M. Committee B-4 on Electrical-Heating, Electrical-Resistance, and Electric-Furnace Alloys. Published as information, June, 1942.

In view of the many applications for these alloys in electric furnace work, consideration is being given to covering this field by means of two or more specifications of more limited scope.

	Grade A1	Grade A2	Grade A3
Tensile strength, min., psi.....	80 000	80 000	80 000
Elongation in 2 in., min., per cent.....................	12	8	4

(*b*) The cross-head speed of the testing machine shall not exceed 1 in. per min.

NOTE.—A discussion of the purpose of the aging test appears in the Appendix.



Pour through head; cover molten head with powdered charcoal, coke dust, etc., immediately after pouring, in order to keep head fluid as long as possible.

FIG. 1.—Test Block for Tension Test Specimen.

Magnetic Permeability

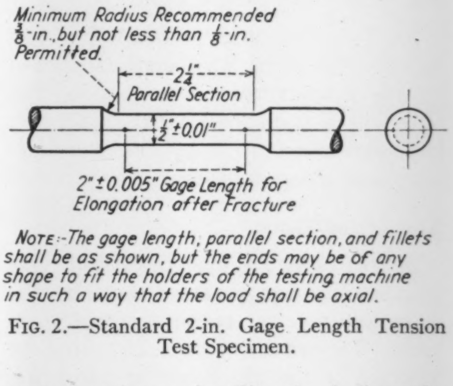
8. When the test specimens are subjected to a magnetizing force of 200 oersteds, the magnetic permeability shall conform to the following requirements:

	Grade A1	Grade A2	Grade A3
Magnetic permeability, max...	1.60	1.35	1.05

NOTE.—The magnetic permeability test gives an approximate measure of the ferrite content and therefore, for the purpose of these specifications, the probable creep strength at 1800 F. (see Appendix). At the present time, the magnetic permeability test for high-temperature alloys is not fully developed. Further work is being done in completing some details of procedure and in the correlation of physical and magnetic properties of these alloys. In the meantime, any use of this test in purchase specifications should be governed by agreement between the manufacturer and the purchaser.

Test Specimens

9. (*a*) The aging test specimens may be taken from test coupons separately cast, as shown in Fig. 1, during the pouring of the melt, or may be taken from the castings. A sufficient number of test coupons shall be provided by the manufacturer for the specified tests. In the case of castings for unusual or severe service, and if so specified in the order, the test coupons shall be attached to the castings at a convenient location. The test specimens shall be machined



Minimum Radius Recommended ⅜-in., but not less than ⅛-in. Permitted.
2¼"
Parallel Section
½" ± 0.01"
2" ± 0.005" Gage Length for Elongation after Fracture

NOTE:-The gage length, parallel section, and fillets shall be as shown, but the ends may be of any shape to fit the holders of the testing machine in such a way that the load shall be axial.

FIG. 2.—Standard 2-in. Gage Length Tension Test Specimen.

to the form and dimensions shown in Fig. 2.

(*b*) The magnetic permeability test specimens shall be taken from test coupons or castings similar to those used for obtaining the aging test specimens. The cast or machined specimens shall be heated in air to 2000 ± 25 F., held within this range for 24 hr., and then quenched in water. After quenching, all scale and superficial oxidized metal shall be completely removed by lightly grinding with a fine-grained wheel in order to avoid errors that might arise from the presence of magnetic oxides formed during heating. The finished specimen shall be cylindrical, not less than 1 in. in length, and the ratio of length to diameter shall be not less than 4. The maximum diameter shall be not greater than the minimum by more than 5 per cent.

Magnetic Permeability Test

10. The magnetic permeability test may be made by any method chosen by the manufacturer. In case of dispute, check tests shall be made by any method agreed upon by the manufacturer and the purchaser.

Number of Tests

11. (*a*) One aging test and one permeability test shall be made by the manufacturer on material from each heat. The results of these tests shall be reported to the purchaser or his representative.

(*b*) If any aging test specimen shows defective machining or develops flaws, it may be discarded and another specimen from the same heat substituted.

(*c*) If any part of the fracture in any aging test specimen is more than $\frac{3}{4}$ in. from the center of the gage length, as indicated by scribe scratches marked on the specimen before testing, another specimen shall be substituted.

Retests

12. (*a*) If the tensile strength or elongation fail to conform to the requirements specified in Section 7, two additional specimens may be tested, both of which shall conform to the requirements specified in Section 7.

(*b*) If the results of the magnetic permeability test do not conform to the requirements specified in Section 8, three additional specimens may be tested. At least two of these shall conform to the requirements specified in Section 8.

Workmanship

13. The castings shall be sound and shall conform substantially to the dimensions on patterns and drawings submitted by the purchaser.

Finish

14. (*a*) The castings shall be free from blow holes, sand inclusions, or other defects which might impair their strength or usefulness.

(*b*) *Repair by Welding.*—Defects which will not impair the effectiveness of the castings may be welded by an approved process. The defects shall be cleaned out to solid metal before welding and, when so required by the inspector, the castings shall be submitted to him in this condition for his approval. Repaired castings shall be heat-treated in accordance with the procedure agreed upon by the manufacturer and the purchaser.

Inspection

15. The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the material ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the

material is being furnished in accordance with these specifications. All tests (except check analysis) and inspection shall be made at the place of manufacture prior to shipment, unless otherwise specified, and shall be so conducted as not to interfere unnecessarily with the operation of the works.

APPENDIX

From the following it will be evident that the aging test and the magnetic permeability test do not provide data for engineering design but are used to classify castings as covered by these specifications with respect to their probable relative reactions to prolonged exposure to usual working temperatures.

A1. *Limiting Creep Stress.*—The probable limiting creep stress values for the three grades of alloys covered by these specifications are shown in the following table:

Grade	Probable Limiting Creep Stress,[a] psi.
A1	1000
A2	1450
A3	1700

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[a] The values shown are for minimum creep rate of 1 per cent in 10,000 hr. at 1800 F.

A2. *Aging Test.*—The purpose of the aging test is to disclose the tendency of the material to decrease in ductility with time at working temperatures. The following general statements may be made concerning the different grades.

(*a*) *Grade A1* may be partially ferritic and subject to some sigma phase development under certain conditions of extended exposure such as at 1200 to 1600 F.

(*b*) *Grade A2*, may be slightly ferritic, that is, it is not necessarily completely austenitic, and even if it should be, is not necessarily permanently so.

(*c*) *Grade A3* is fully austenitic as cast and may be expected to remain substantially so in service in the usual temperature ranges.

(*d*) The elongation values specified are for "as cast" test specimens after an aging treatment at the time and temperature stated. The elongation for grades A1 and A2, especially for grade A1, may drop below that for grade A3 after a more extended exposure at intermediate temperatures between 1200 and 1600 F.

A3. *Magnetic Permeability Test.*—The purpose of the magnetic permeability test is to provide a measure of the tendency of the alloy to develop ferrite and therefore low creep strength. The time and temperature for the conditioning treatment have been chosen in the light of experiments as those giving the largest ferritizing effect under reasonable test conditions.

The low creep strength associated with the ferritic alloys covered by these specifications apparently arises from the fact that the ferritic phase is much weaker at the higher working temperatures than is the austenitic. Experimental work also has shown a correlation between creep strength and ferrite content as measured by magnetic permeability.

APPENDIX II

PROPOSED METHOD OF TEST FOR EFFECT OF CONTROLLED ATMOSPHERES UPON ALLOYS IN ELECTRIC FURNACES[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia, Pa.

Scope

1. This method of test is intended for determining the following:

(*a*) The action of commercial protective atmospheres upon heat-resisting alloys such as are used in industrial furnaces for electric-heating elements and other parts intended to operate at high temperatures,

(*b*) The temperature at which attack occurs, and

(*c*) The effect of stress upon the rate of attack.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Electric Furnace.*—A vertical electric tube furnace with the heating unit wound on an alundum tube and so distributed as to be capable of maintaining a temperature of 2000 F. across the mid-section of the tube and a smooth downward gradient to 1000 F. or less at each end. The furnace casing shall be made of steel, and shall be made gas tight either by welding or by the use of gaskets. Special care shall be taken to arrange the terminals and other joints so that there will be no perceptible leakage under pressures up to 3 in. of water. Means shall be provided for introducing gas at the top of the tube and exhausting it at the bottom. A water leg shall be provided to catch any moisture driven off during starting up, or when excessive moisture is inadvertently introduced with the atmosphere. Two closed-end, thin-walled, metal protecting tubes shall be sealed through the top, one to be used for temperature control with the control couple tip located at the mid-point of the furnace chamber length, and the other to be used for exploring the temperature distribution throughout the length of the test chamber.

NOTE.—Details of furnaces that have been found suitable are shown in Figs. 1 and 2.

(*b*) *Gas Flow Meter.*—A gas flow meter capable of measuring at normal temperature and pressure, gas flows as low as ½ cu.ft. per hr. within plus or minus 10 per cent.

(*c*) *Control Instrument.*—Any standard temperature control instrument together with thermocouple capable of controlling temperature at 2000 F. will be satisfactory.

Test Specimen

3. (*a*) The area of exposed surface per unit length of the test specimen should preferably be large compared with that of its cross-section. Specimens 0.375 in. in

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee B-4 on Electrical-Heating, Electrical-Resistance, and Electric-Furnace Alloys. Published as information, June, 1942.

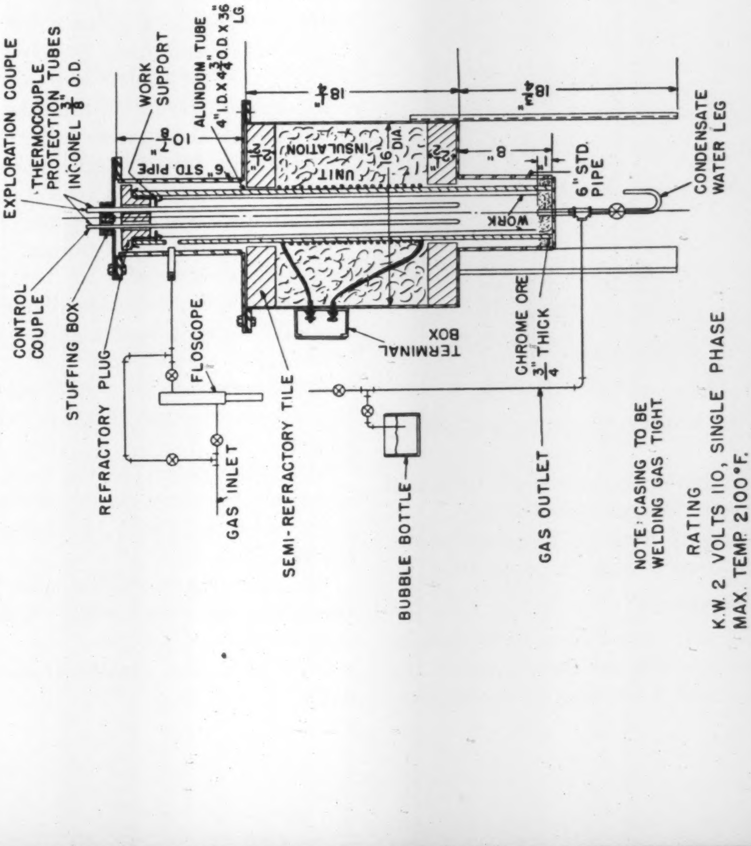
EXPLORATION COUPLE
THERMOCOUPLE PROTECTION TUBES INCONEL 3/8" O.D.
WORK SUPPORT
ALUNDUM TUBE 4" I.D. X 4 3/4" O.D. X 36" LG.
10 7/8"
6" STD. PIPE
18 1/4"
18 1/4"
INSULATION
UNIT
16" DIA.
2 1/2"
2 1/2"
8"
1"
6" STD. PIPE
WORK
CONDENSATE WATER LEG
CONTROL COUPLE
STUFFING BOX
REFRACTORY PLUG
FLOSCOPE
GAS INLET
SEMI-REFRACTORY TILE
TERMINAL BOX
CHROME ORE 3/4" THICK
BUBBLE BOTTLE
GAS OUTLET
NOTE: CASING TO BE WELDING GAS TIGHT
RATING
K.W. 2 VOLTS 110, SINGLE PHASE
MAX. TEMP. 2100°F.

FIG. 1.—Outline of Electric Tube Furnace.

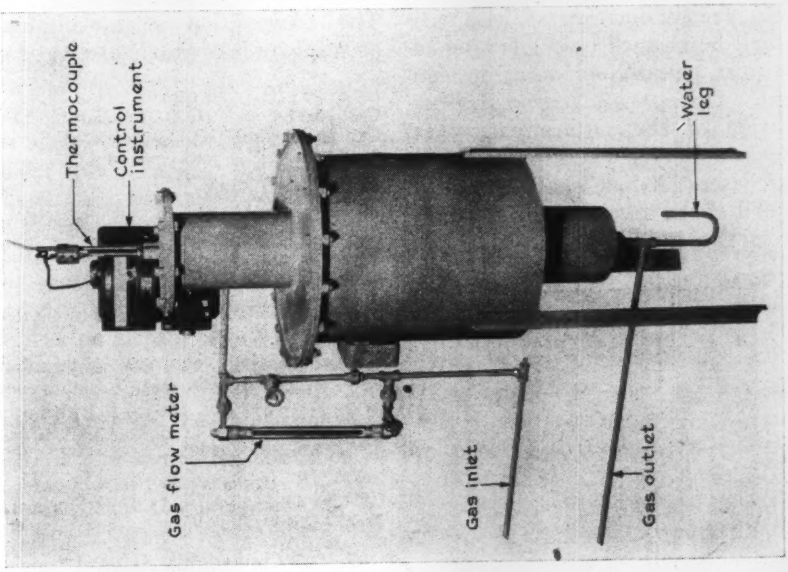
Thermocouple
Control instrument
Water leg
Gas flow meter
Gas inlet
Gas outlet

FIG. 2.—Electric Tube Furnace.

width by 0.035 in. in thickness by 25 in. in length are satisfactory. The specimens shall be cleaned before testing to remove dirt, grease, or other foreign material. There shall be no further preparation of the surface except as agreed upon by the manufacturer and the purchaser. A hole shall be drilled in one end of the specimen for suspending it in the furnace.

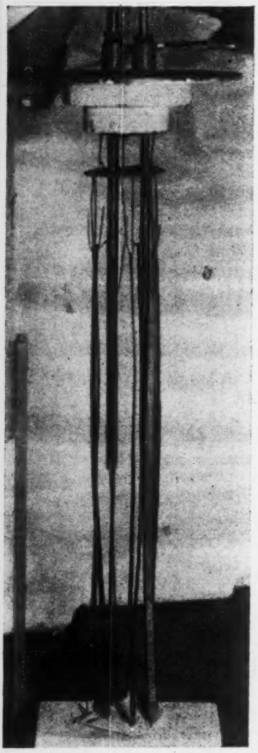

FIG. 3.—Method of Suspending Specimens for Test.

(*b*) Several specimens shall be tested at the same time, as shown in Fig. 3.

(*c*) Weights may be suspended from the specimens to introduce stress.

Test Atmosphere

4. The test atmosphere used shall be as agreed upon by the manufacturer and the purchaser, according to the application for which the alloy is to be used. The composition of a suitable test atmosphere is approximately as follows:

Hydrogen (H_2), per cent	16
Carbon Monoxide (CO), per cent	10
Carbon Dioxide (CO_2), per cent	4
Methane[a] (CH_4), max., per cent	1
Moisture (H_2O), per cent	1.5 to 2.5
Sulfur	not over 2 grains per 100 cu. ft.
Nitrogen (N_2), per cent	remainder

[a] Also includes hydrocarbon.

NOTE.—This atmosphere may be obtained from:

(*a*) Burning coke gas with sulfur low enough to meet the above analysis (530 Btu. per cu. ft.) with a ratio of 2½ parts of air to 1 part of gas, or

(*b*) Natural gas burned with a ratio of 5½ parts of air to 1 part of gas.

Procedure

5. (*a*) Thoroughly dry out the furnace by operating at the test temperature for several days, and then pass the gas to be tested through the furnace for at least 24 hr. before removing the power. The furnace shall be cooled to 1000 F. or less with the gas passing through the furnace.

(*b*) Install the specimens to be tested, and seal the furnace tight. Adjust the gas flow to correspond to a mean linear velocity through the tube and over the work as calculated for normal temperature and pressure of 5 to 7 in. per min. In a tube 4 in. in inside diameter, this equals 2.16 to 3.03 cu.ft. of gas per hour at normal temperature and pressure. The temperature of the furnace when the specimens are installed shall not exceed 1000 F.

(*c*) Apply power to the furnace and stabilize the temperature of the maximum point at 2000 F. avoiding swings beyond this.

(*d*) Take an exploration curve in check thermocouple tube, while maintaining constant flow of gas.

(*e*) Check the control thermocouple daily.

(*f*) Maintain conditions constant for approximately 300 hr.

(*g*) Remove power and cool furnace while uniform gas flow is maintained through the tube.

(*h*) Inspect the specimens. If there has been any attack, it may be shown by an enlarged section, or by blisters, or possibly by both. Wherever attack occurs, the attack material is magnetic. If the specimen is broken at the point of attack, the fracture will appear oxidized to the depth of the attack, although a file or saw cut will appear bright. The attack usually occurs at or near the point on the specimen where the temperature during the test was 1750 F. There is decreasing attack above and below this temperature.

Precautions

6. The rate of gas flow is very important as it has been established that a flow of 10 cu. ft. per hr. with a tube 4 in. in diameter will not attack the most susceptible alloy. Also, a flow of 0.5 cu. ft. per hr. under the same conditions will not cause any attack. It is important that sulfur be kept very low since appreciable amounts may be the cause of misleading conclusions, as the sulfur attack may occur at approximately 1750 F. The sulfur contained in ordinary city gas is usually not sufficient to cause attack. A dew point of 60 to 70 F. appears to be satisfactory.

Report

7. The report shall include the following:

(*1*) Grade of material,

(*2*) Maximum temperature and time of exposure,

(*3*) Temperature distribution in furnace,

(*4*) Nature of attack (nodular, penetrating, etc.),

(*5*) Location of attack with reference to temperature scale,

(*6*) Extent of region showing increased magnetizability, with reference to temperature scale,

(*7*) Gas analysis, including carbon monoxide, etc.,

(*8*) Load in pounds per square inch, if any load is applied in the test, and

(*9*) Microsection.

REPORT OF COMMITTEE B-5

ON

COPPER AND COPPER ALLOYS, CAST AND WROUGHT

Committee B-5 on Copper and Copper alloys held three meetings during the year: in Chicago, Ill., on June 24, 1941; in Washington, D. C., on November 6 and 7, 1941, and in New York, N. Y., on January 27, 1942, with an attendance at the several sessions ranging from 20 to 40 members.

The following additions and changes in membership have occurred:

Chairman, C. H. Greenall—new affiliation: Major, Ordnance Department, Frankford Arsenal.
Subcommittee IV—E. S. Bunn replaces L. A. Ward, resigned.
Subcommittee V—H. H. Stout, Jr., replaces W. H. Bassett, Jr., resigned.
Subcommittee VII—C. A. Hill appointed chairman, replacing R. S. Pratt, resigned.
Subcommittee XII—C. S. Cole, Consultant, War Production Board; new personal member, retains Chairmanship.
Subcommittee XIII—J. E. McGraw, replaces R. G. Thompson, resigned.

New Members:

D. K. Crampton, representing Kennecott Copper Corp.
R. J. Allen (Worthington Pump and Machinery Corp.)
H. A. Gillan (Eastman Kodak Co.)
R. A. Frye, additional representative of Westinghouse Electric and Manufacturing Co.
L. A. Wenn, representing International Business Machines Corp.
H. A. Marchant, representing Chrysler Corp.
W. P. Eddy, Jr. (Yellow Truck and Coach Mfg. Co.)
R. W. Orr, representing RCA Manufacturing Co.
E. A. Basilio, consulting member (General Railway Signal Corp.).
Curtis Pigott, representing Phelps Dodge Refining Corp.

New Representatives:

H. C. Ashley, replacing L. A. Ward, Chase Brass and Copper Co.
W. F. Aylard, replacing H. P. Croft, Chase Brass and Copper Co.
David Basch, replacing J. M. Sampson, General Electric Co.
J. T. Jarman, replacing H. J. Stein, Allis-Chalmers Mfg. Co.
C. J. Snyder, replacing W. H. Bassett, Jr., Anaconda Wire and Cable Co.
G. A. Cullen, replacing L. A. Wenn, International Business Machines Corp.
G. R. Gohn, replacing C. H. Greenall, Bell Telephone Laboratories and dropping personal membership.
A. I. Heim, replacing C. S. Cole, Copper and Brass Research Assn.
Major C. H. Greenall, as additional representative with Lt. Col. L. S. Fletcher of U. S. Army Ordnance Dept., Frankford Arsenal.

Resigned from the Society:

C. E. Margerum, U. S. Navy, Bureau of Ordnance
H. A. Gillan, Eastman Kodak Co.
J. B. Neiman, Am. Smelting & Refining Co.

Deceased:

J. M. Sampson, General Electric Co.
H. A. Staples, Phelps Dodge Copper Products Corp.
H. J. Stein, Allis-Chalmers Manufacturing Co.

In accordance with action taken at the January 27, 1942, meeting, those members who have entered Government service will have their names placed on the Inactive List unless they specifically request active membership. At the present time, this list includes the names of W. H. Bassett, Jr., and H. P. Croft.

The committee lost by death, one long-standing member, Mr. H. A. Staples who gave freely of his time and experience to committee affairs for many years. The committee has also lost the service of Messrs. J. M. Sampson and H. J. Stein who were valued members of the committee.

At the present time the committee consists of 107 members of which 97 are voting members; 39 are classified as producers, 41 as consumers, and 17 as general interest members.

Subsequent to the annual meeting, Committee B-5 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative Specifications for:

Phosphor Bronze Rods, Bars, and Shapes (B 139 - 41 T),
Phosphor Bronze Wire (B 159 - 41 T), and

Revision of:

Tentative Specifications for Beryllium-Copper Alloy Bars, Rods, Sheet, Strip, and Wire (B 120 - 41 T).

These recommendations were accepted[1] by Committee E-10 on August 25, 1941, and the new and revised tentative standards assigned the A.S.T.M. serial designations indicated above, appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part I, pp. 427, 435, and 475.

On November 8, 1941, Committee E-10 accepted[1] a tentative revision proposed by Committee B-5 in the Standard Specifications for Phosphor Bronze Sheet and Strip (B 103 - 40) which appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 595.

On November 28, 1941, Committee E-10 accepted[1] new Tentative Specifications for Aluminum Bronze Sheet and Strip (B 169 - 41 T), submitted by Committee B-5, which appear in the special compilation of "A.S.T.M. Standards on Copper and Copper Alloys," 1941, p. 305, and are also available as a separate reprint.

On April 1, 1942, Committee E-10 accepted[1] new Tentative Specifications for Copper-Alloy Condenser Tube Plates (B 171 - 42 T), submitted by Committee B-5, which are available as a separate reprint.

EMERGENCY ALTERNATE PROVISIONS

In the interest of conserving tin and also to conserve the use of new copper and to permit the use of scrap metal, Emergency Alternate Provisions in the following specifications were submitted by Committee B-5 to Committee E-10 on Standards:

Tentative Specifications for:

Copper-Base Alloys in Ingot Form for Sand Castings (B 30 - 41 T),
Tin-Bronze and Leaded Tin-Bronze Sand Castings (B 143 - 41 T),
High-Leaded Tin-Bronze Sand Castings (B 144 - 41 T),
Leaded Red Brass and Leaded Semi Red Brass Sand Castings (B 145 - 41 T),
Aluminum-Bronze Sand Castings (B 148 - 41 T),

Standard Specifications for:

Castings of the Alloy: Copper 88 per cent, Tin 8 per cent, Zinc 4 per cent (B 60 - 41), and
Composition Brass or Ounce Metal Castings (B 62 - 41).

These Emergency Alternate Provisions were accepted by Committee E-10 on April 6, 1942. Upon further recommendation of Committee B-5, additional Emergency Alternate Provisions in the above specifications were approved by Committee E-10 on May 26, 1942. The alternate provisions approved in April as well as those accepted in May, have been published as stickers for attachment to the specifications to which they apply, under the following designations:

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee B-5 reported the following results of the letter ballot vote from a committee membership of 88: B 139 and B 159, 70 ballots returned, affirmative 52, negative 0, ballots marked "not voting" 18; B 120, 66 ballots returned, affirmative 57, negative 1, ballots marked "not voting" 8. The following results of the letter ballot vote were reported by Committee B-5 from a membership of 96: B 103, 56 ballots returned, affirmative 44, negative 0, ballots marked "not voting" 12; B 169, 53 ballots returned, affirmative 35, negative 3, ballots marked "not voting" 15; B 171, 61 ballots returned, affirmative 39, negative 0, ballots marked "not voting" 22.

EA-B 30	EA-B 148
EA-B 143	EA-B 60
EA-B 144	EA-B 62
EA-B 145	

Committee B-5 has also submitted proposed Emergency Alternate Provisions in the Tentative Specifications for Leaded Yellow Brass Sand Castings for General Purposes (B 146 - 41 T) in order to make possible the use of yellow brass scrap which is available in fairly large tonnage from the ingot producers. These Emergency Alternate Provisions EA-B 146 were accepted by Committee E-10 on May 26, 1942, and have been published in the form of a sticker for attachment to the specifications.

At several meetings, the use of fire-refined copper was discussed with the result that Emergency Alternate Provisions in a number of the specifications under the jurisdiction of Committee B-5 have been approved and are to be presented to the Society upon completion by Committee B-2 on Non-Ferrous Metals and Alloys of the specification requirements for this type of copper.[2]

RECOMMENDATIONS AFFECTING STANDARDS

As a result of the year's work, the committee is recommending revisions of eighteen tentative standards, revisions in seven standards for immediate adoption, the adoption as standard of two tentative standards and tentative revision in one standard, and the withdrawal of one standard which has been replaced by new tentative specifications. The standards and tentative standard affected are listed in Table I. The recommendations being presented are included in detail in Appendix I.

The recommendations in this report have been submitted to letter ballot of the committee which consists of 97 voting members, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. REVISIONS OF TENTATIVE STANDARDS			
Spec. for Cartridge Brass Sheet, Strip, and Disks (B 19 - 41 T)	48	0	24
Spec. for Naval Brass Rods (B 21 - 40 T)	51	0	11
Spec. for Copper-Base Alloys in Ingot Form for Sand Castings (B 30 - 41 T)	53	0	15
Spec. for Seamless Copper Tubing, Bright Annealed (B 68 - 41 T)	43	0	19
Spec. for Copper-Base Alloy Forging Rods, Bars, and Shapes (B 124 - 41 T)	51	0	21
Spec. for Copper Rods, Bars, and Shapes (B 133 - 40 T)	49	0	13
Spec. for Brass Wire (B 134 - 41 T)	51	0	11
Spec. for Miscellaneous Brass Tubes (B 135 - 40 T)	44	0	18
Spec. for Phosphor Bronze Bars, Rods, and Shapes (B 139 - 41 T)	51	0	11
Spec. for Tin-Bronze and Leaded Tin-Bronze Sand Castings (B 143 - 41 T)	55	0	17
Spec. for High-Leaded Tin-Bronze Sand Castings (B 144 - 41 T)	41	2	19
Spec. for Leaded Red Brass and Leaded Semi Red Brass Sand Castings (B 145 - 41 T)	53	0	15
Spec. for Leaded Yellow Brass Sand Castings for General Purposes (B 146 - 41 T)	42	2	18
Spec. for High-Strength Yellow Brass and High-Strength Leaded Yellow Brass Sand Castings (B 147 - 41 T)	41	2	19
Spec. for Aluminum-Bronze Sand Castings (B 148 - 41 T)	42	2	18
Spec. for Leaded Nickel-Brass (Leaded Nickel-Silver) and Leaded Nickel-Bronze (Leaded Nickel-Silver) Sand Castings (B 149 - 41 T)	41	2	19
Spec. for Copper-Nickel-Zinc Alloy Rods and Wire (B 151 - 41 T)	51	0	11
Spec. for Phosphor Bronze Wire (B 159 - 41 T)	51	0	11
II. REVISIONS OF STANDARDS, IMMEDIATE ADOPTION			
Spec. for Copper Bars for Locomotive Staybolts (B 12 - 33)	51	0	11
Spec. for Free-Cutting Brass Rod for Use in Screw Machines (B 16 - 41; ASA: H8-1941)	56	0	16
Spec. for Brass Pipe, Standard Sizes (B 43 - 41)	44	0	18
Spec. for Steam or Valve Bronze Castings (B 61 - 41)	55	0	17
Spec. for Copper-Silicon Alloy Rods, Bars, and Shapes (B 98 - 41)	47	1	14
Spec. for Copper-Silicon Alloy Wire for General Purposes (B 99 - 41)	51	0	11
Spec. for Phosphor Bronze Sheet and Strip (B 103 - 40)	35	0	27
III. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Spec. for Copper-Silicon Alloy Plate and Sheet for Pressure Vessels (B 96 - 41 T)	38	0	24
Spec. for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 - 40 T)	42	1	18
IV. ADOPTION OF TENTATIVE REVISION OF STANDARD			
Spec. for Phosphor Bronze Sheet and Strip (B 103 - 40)	35	0	27
V. WITHDRAWAL OF STANDARD			
Spec. for Muntz Metal Condenser Tube Plates (B 57 - 27)	49	0	13

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[2] See Editorial Note, p. 190.

ACTIVITIES OF SUBCOMMITTEES

Committee B-5 during the past year has continued the plan of completing new work as far as possible in subcommittees or in individual subgroups. As a result, better progress than usual has been achieved and many details have been cleared through Committee E-10 on Standards.

Subcommittee I on Copper-Zinc Sheet and Strip (G. H. Harnden, chairman) has continued the grain size round-robin study. Preliminary work has been completed and the data are now undergoing statistical analysis.

The special Subcommittee on Primer Brass is continuing the investigation but a final decision as to methods of testing has not as yet been reached. The chairman has presented for the consideration of the subcommittees having jurisdiction over sheet and strip metal a table giving suggested preferred sizes for the purchase of such material. The table has been set up on the basis of the 20-series of the American Standard Preferred Numbers. The subcommittee is recommending minor changes in two specifications as given in Appendix I.

Subcommittee II on Copper-Tin Sheet and Strip (H. V. Schlacks, chairman) has cooperated with the main committee in preparing several minor changes, mostly of an editorial nature, in the specifications under its jurisdiction. Items subject to letter ballot have been approved and appear in Appendix I.

Subcommittee III on Copper-Nickel-Zinc Sheet and Strip (E. S. Bunn, chairman) has approved revisions in tolerances as recommended by Subcommittee XIII and has made slight editorial changes in the specifications under its jurisdiction.

Subcommittee IV on Miscellaneous Copper-Base Sheet and Strip Alloys (L. A. Ward, chairman) has held special meetings on matters pertaining to beryllium copper and the copper-silicon alloys resulting in a revision of the Tentative Specifications for Beryllium-Copper Alloy Bars, Rods, Sheet, Strip, and Wire (B 120 – 41 T) which were accepted by Committee E-10 as noted earlier in this report.

New Tentative Specifications for Aluminum Bronze Sheet and Strip (B 169 – 41 T) were also approved and submitted to the Society for publication through Committee E-10. Changes in tolerances and other changes, minor in character, in other specifications have been approved as reported earlier. The Tentative Specifications for Copper-Silicon Alloy Plate and Sheet for Pressure Vessels (B 96 – 41 T) are recommended for adoption as standard.

Because of a change in duties, Mr. Ward felt it necessary to resign the chairmanship late in 1941 but kindly carried along the duties of the position until the appointment of his successor, Mr. E. S. Bunn, on February 17, 1942.

Subcommittee V on Miscellaneous Copper-Base Wire and Rod Alloys (W. H. Bassett, Jr., chairman) completed the new Tentative Specifications for Phosphor Bronze Rod (B 139 – 41 T) and for Phosphor Bronze Wire (B 159–41 T) which were accepted by Committee E-10. Meetings were held in Washington and in New York at which the physical test requirements and tolerances in all of the specifications under its jurisdiction were carefully reviewed. Approved changes are given in Appendix I. There is much active work on hand, however, most of which should be concluded before the annual meeting.

A special subcommittee has been named to prepare a new specification for bus bar copper in all commercially available forms. Another special subcommittee has been named to divide the Tentative Specifications for Beryllium-Copper Alloy (B 120 – 41 T) into two distinct specifications, one for rod and wire and the other for sheet and strip. Still another special subcommittee has been named to prepare tentative specifications for rods for use as valve stems.

The chairman resigned as of February 1, 1942, because of his new duties in the Ordnance Department, Washington, D. C., Mr. H. H. Stout, Jr., who has been very active in the affairs of the subcommittee was appointed as the new chairman.

Subcommittee VI on Condenser Tubes (G. C. Holder, chairman) prepared new Tentative Specifications for Copper-Alloy Condenser Tube Plates (B 171 – 42 T) which were accepted by Commitmittee E-10 on Standards on April 1, 1942. The subcommittee recommends the adoption as standard of the Tentative Specifications for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 – 40 T).

Subcommittee VII on Copper or Deoxidized Copper Tubes (R. S. Pratt, chairman) has recommended certain revisions in the specifications under its jurisdiction, all of which are given in Appendix I. These changes pertain principally to the simplification of paragraphs on the methods of testing.

The chairman resigned as of March 17, 1942, because of the extra burden of new duties in the new Bridgeport Brass Co. plant at Indianapolis. Mr. C. A. Hill, of Mueller Brass Co., who has been a very valued member of the subcommittee was appointed as the new chairman on April 2, 1942.

Subcommittee VIII on Copper Alloy Tubes for General Use (Alan Morris, chairman) has recommended changes similar to those offered by Subcommittee VII relating to the revision and clarification of sections on methods of test in its specifications as given in Appendix I.

Subcommittee IX on Copper-Base Alloy Forgings (J. J. Kanter, chairman) has recommended changes in alloy composition in the Tentative Specifications for Copper-Base Alloy Forging Rods, Bars, and Shapes (B 124 – 41 T) as indicated in Appendix I.

Subcommittee X on Copper-Base Alloys or Sand Castings (G. H. Clamer, chairman) considered in detail some 13 items included on its agenda for the meeting held on November 7 in Washington. Matters subject to action by the main committee are listed in Table I and given in detail in Appendix I. Basis of purchase paragraphs and a section on certification of composition have been inserted in most of the specifications for sand castings. One of the principal items for discussion was a suggestion for the reduction in the number of alloys and alloy specifications for Government use, particularly during the present National Emergency. A joint committee has been sponsored under the W.P.B. and this is now actively engaged on the problem.

The paper by A. J. Smith and J. W. Bolton, on "Effects of Sulfur and Antimony on Steam or Valve Bronze Castings"[3] describes important studies of bronze castings covered by specifications prepared by this subcommittee.

Because of the illness of the chairman, the meeting to be held in New York on January 27, was cancelled. However, to assist the chairman, the advisory Committee of Committee B-5 requested Second Vice-Chairman J. J. Kanter to carry on the general program of the subcommittee, particularly with regard to the development of Emergency Specifications for bronze casting alloys that would conserve tin. The Specifications Branch, Bureau of Industrial

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[3] ASTM BULLETIN, No. 116, May, 1942, p. 18.

Conservation, through C. S. Cole has aided in this work which resulted in the preparation of Emergency Alternate Provisions in eight specifications for brass and bronze castings as listed earlier in this report.

Through cooperation with the American Foundrymen's Association there are in preparation A.F.A. Recommended Practice instructions which will embrace the new A.S.T.M. specifications for brass and bronze castings prepared by this subcommittee. The first of these recommended practices will cover the tin bronze castings.

Specifications for silicon bronze castings are in preparation and it is expected that they will be acted on by the subcommittee at its June meeting.

Much interesting information has been developed on the subject of test bars and test bar practice. This data and information have been compiled by J. J. Curran, J. W. Bolton, F. L. Wolf, and W. G. Martin and are presented in a report on "Studies of Machined *versus* As-Cast Test Specimens for Copper-Alloy Sand Castings," which appears in Appendix II.

Subcommittee XI on Methods of Test for Copper and Copper Alloys (A. J. Phillips, chairman) has given special attention during the year to methods of determining yield strength in copper-base alloys under the jurisdiction of Committee B-5. In spite of the activity of the subcommittee, final decisions have not as yet been reached as to the preferred procedure. Progress was reported by the special subcommittee on the tension testing of thin strips and by another special committee on compression testing.

Subcommittee XII on Publication of General Information (C. S. Cole, chairman) concluded its work in editing the new compilation of "A.S.T.M. Standards on Copper and Copper Alloys," issued in December, 1941. This new volume has within two months taken a position among the "required" publications of the Society. The volume contains 73 specifications, including all of the specifications under the jurisdiction of Committee B-5, also those under Committee B-1 on Copper and Copper-Alloy Wire for Electrical Conductors, several under Committee B-2 on Non-Ferrous Metals and Alloys, and all pertinent methods of test. The subcommittee has also been of great assistance in the formulation of "basis of purchase" clauses for most of the specifications for copper alloy.

Although the chairman has been called to service in Washington with the Bureau of Industrial Conservation, his continuation in office has been approved by the Advisory Committee.

Subcommittee XIII on Tolerances (R. G. Thompson, chairman) has been one of the most active subcommittees because it is employed in a new and active field. It has been of the greatest service to all of the other subcommittees and has relieved them of much detail and duplication of effort in the formulation and approval of tolerances. The subcommittee has maintained close contact with the Standards Committee of the Copper and Brass Research Assn. and has given to the Association the consumers' point of view. J. E. McGraw, General Electric Co. has been appointed the new chairman in place of R. G. Thompson resigned.

Special Subcommittee on Types of Copper (H. H. Stout, Jr., chairman) has prepared several drafts of a proposed tentative classification of commercial coppers. These have been presented to the Advisory Committee for comments and approval for submittal of the data to Committee B-2. When substantial agreement is reached, the proposed classification is to be forwarded to Committee B-2 with the request that it develop and publish if possible requirements comparable to those suggested by the special subcommittee. When and if the classification for commercial coppers is published as tentative under an A.S.T.M. designation, Committee B-5 will then have to reconsider all designations for copper in existing specifications under its jurisdiction.

The present officers of the committee have been re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 97 voting members; 68 members returned their ballots, of whom 63 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

C. H. GREENALL,
Chairman.

C. H. DAVIS,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee B-5 presented to the Society through Committee E-10 on Standards the following recommendations:

Revisions of Tentative Specifications for:

Phosphor Bronze Rods, Bars, and Shapes (B 139 – 41 T), and
Phosphor Bronze Wire (B 159 – 41 T).

These recommendations were accepted by Committee E-10 on August 24, 1942, and the revised tentative specifications appear in the 1942 Book of A.S.T.M. Standards, Part I, pp. 1280 and 1293.

Committee B-5 also submitted to Committee E-10 the following recommendations which were accepted on December 11, 1942:

Revisions of Tentative Specifications for:

Cartridge Brass Sheet, Strip, and Disks (B 19 – 42 T),
Bronze Castings for Turntables and Movable Bridges and for Bearing and Expansion Plates of Fixed Bridges (B 22 – 40 T),
Brass Sheet and Strip (B 36 – 41 T),
Leaded Brass Sheet and Strip (B 121 – 41 T),
Copper-Nickel-Zinc and Copper-Nickel Alloy Sheet and Strip (B 122 – 39 T),
Copper-Base Alloy Forging Rods, Bars, and Shapes (B 124 – 42 T),
Cartridge Brass Cartridge Case Cups (B 129 – 40 T),
Gilding Metal Sheet and Strip (B 130 – 41 T),
Gilding Metal Bullet Jacket Cups (B 131 – 40 T),
Miscellaneous Brass Tubes (B 135 – 42 T),
Aluminum-Bronze Sand Castings (B 148 – 42 T),
Aluminum Bronze Sheet and Strip (B 169 – 41 T), and
Copper-Alloy Condenser Tube Plates (B 171 – 42 T).

The revised tentative specifications appear in the 1942 Book of A.S.T.M. Standards, Part I, pp. 1216, 1322, 1203, 1210, 1235, 1264, 1222, 1226, 1231, 1312 1352, 1241, and 1251.

On December 14, 1942, the following Emergency Alternate Provisions, submitted by Committee B-5, were accepted by Committee E-10:

DESIGNATION	EMERGENCY ALTERNATE PROVISIONS IN
	Standard Specifications for:
EA – B 12	Copper Bars for Locomotive Staybolts (B 12 – 42)
EA – B 100	Rolled Copper-Alloy Bearing and Expansion Plates for Bridge and Other Structural Uses (B 100 – 40)
EA – B 103	Phosphor Bronze Sheet and Strip (B 103 – 42)
EA – B 111	Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 – 42)

DESIGNATION	EMERGENCY ALTERNATE PROVISIONS IN
	Tentative Specifications for:
EA - B 19	Cartridge Brass Sheet, Strip, and Disks (B 19 - 42 T)
EA - B 22	Bronze Castings for Turntables and Movable Bridges and for Bearing and Expansion Plates of Fixed Bridges (B 22 - 42 T)
EA - B 36	Brass Sheet and Strip (B 36 - 42 T)
EA - B 121	Leaded Brass Sheet and Strip (B 121 - 42 T)
EA - B 122	Copper-Nickel-Zinc and Copper-Nickel Alloy Sheet and Strip (B 122 - 42 T)
EA - B 124	Copper-Base Alloy Forging Rods, Bars, and Shapes (B 124 - 42 T)
EA - B 129	Cartridge Brass Cartridge Case Cups (B 129 - 42 T)
EA - B 130	Gilding Metal Sheet and Strip (B 130 - 42 T)
EA - B 131	Gilding Metal Bullet Jacket Cups (B 131 - 42 T)
EA - B 133	Copper Rods, Bars, and Shapes (B 133 - 42 T)
EA - B 134	Brass Wire (B 134 - 42 T)
EA - B 135	Miscellaneous Brass Tubes (B 135 - 42 T)
EA - B 139	Phosphor Bronze Rods, Bars, and Shapes (B 139 - 42 T)
EA - B 148a	Aluminum-Bronze Sand Castings (B 148 - 42 T)
EA - B 151	Copper-Nickel-Zinc Alloy Rod and Wire (B 151 - 42 T)
EA - B 152	Copper Sheet, Strip, and Plate (B 152 - 41 T)
EA - B 159	Phosphor Bronze Wire (B 159 - 42 T)
EA - B 169	Aluminum Bronze Sheet and Strip (B 169 - 42 T)
EA - B 171	Copper-Alloy Condenser Tube Plates (B 171 - 42 T)

These emergency provisions were published in ASTM BULLETIN, No. 120, January, 1943, p. 54, and have also been issued in the form of pink stickers for attachment to the standards to which they apply.

APPENDIX I

RECOMMENDATIONS AFFECTING STANDARDS FOR COPPER AND COPPER ALLOYS, CAST AND WROUGHT

In this Appendix are given the recommendations affecting certain standards and tentative standards covering copper and copper alloys, cast and wrought, which are referred to earlier in this report.[1] The standards and tentative standards appear in their present form in either the 1939 Book of A.S.T.M. Standards, Part I or the 1940 or 1941 Supplements, Part I, as indicated by the final number in the A.S.T.M. designation.

REVISIONS OF TENTATIVE STANDARDS

The committee recommends that the following 18 tentative specifications be revised as indicated below and continued as tentative.

Tentative Specifications for Cartridge Brass Sheet, Strip, and Disks (B 19 – 41 T):

Section 14.—Change the second sentence to read as follows by the addition of the italicized word and the omission of the words in brackets:

> The tolerances for sheet *and* strip [and disks] shall be those shown in Table V.

Section 16.—In the table of diameter tolerances, change the values for disks over 3 to 6 and over 6 to 8 in. in diameter to read as follows in order that they will agree with Army Specifications 57-173-B:

Nominal Diameter, in.	Tolerances, in.	
	Plus	Minus
Over 3 to 6, incl.	0.015	0
Over 6 to 8, incl.	0.015	0

Tentative Specifications for Naval Brass Rods, Bars, and Shapes (B 21 – 40 T):

Section 7.—Change to read as follows by the addition of the italicized words and figures:

> 7. *Before testing the specimen shall be properly cleaned.* A 6-in. length of the test specimen, but in no case less than two times its diameter, shall stand, without cracking, an immersion for 15 min. in an aqueous mercurous nitrate solution containing 100 g. of mercurous nitrate and 13 ml. of HNO_3 (sp. gr. 1.42) per liter of solution (Note). *Immediately after removal from the solution, the specimen shall be wiped free of excess mercury and examined.*

New Section.—Add a new Section 10 on Methods of Testing to read as follows, renumbering the subsequent sections accordingly:

> 10. *Methods of Testing.*—The properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

Test	A.S.T.M. Designation[a]
Tensile Strength	E 8
Mercurous Nitrate Test	B 154

[a] These designations refer to the following methods of testing:
Tentative Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8), and
Tentative Method of Mercurous Nitrate Test for Copper and Copper-Alloys (A.S.T.M. Designation: B 154).

Tentative Specifications for Copper-Base Alloys in Ingot Form for Sand Castings (B 30 – 41 T):

Table I.—Omit the nominal composition for aluminum bronze alloy 9A and add the following for alloys 9A-1 and 9A-2:

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[1] See p. 186.

Classification and Numerical Designation		Commercial Designation	Nominal Composition		
			Copper, per cent	Iron, per cent	Aluminum, per cent
Aluminum Bronze	9A-1	Grade A	88.00	3.00	9.00
	9A-2	Grade C	86.00	3.50	10.50

Table II.—Make the following changes in the chemical requirements for alloys 2A and 5A:

Change the iron content of alloy 2A from "0.15 max." to read "0.20 max." per cent.

Change the copper content of alloy 5A from "79.50 min., 81.50 max." to read "78.00 min., 82.00 max." per cent.

Change the lead content of alloy 5A from "6.50 min., 7.50 max.," to read "6.00 min., 8.00 max." per cent.

Change the minimum zinc content of alloy 5A from "8.50" to read "7.00" per cent.

In place of the requirements in Table II for alloy 9A substitute the following requirements for alloys 9A-1 and 9A-2:

Chemical Composition	Alloy 9A-1	Alloy 9A-2
Copper, per cent	86.75 to 88.75	78.00 to 86.00
Aluminum, per cent	8.50 to 9.50	10.30 to 11.20
Iron, per cent	2.75 to 3.25	3.00 to 3.75
Nickel, max., per cent	....	5.00
Manganese, max., per cent	....	3.50
Tin, max., per cent	....	0.20
Total other constituents, max., per cent	0.50	0.50

Tentative Specifications for Seamless Copper Tubing, Bright Annealed (B 68 – 41 T):

Section 8.—Change the table of expansion requirements to read as follows by the addition of the italicized figures and the omission of those in brackets:

Outside Diameter, in.	Expansion of Outside Diameter, per cent
[1] *3/4* and under	40
Over [1] *3/4*	30

Section 12.—Change from its present form to read as follows:

12. *Methods of Testing.*—The properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

Test	A.S.T.M. Designation[a]
Tensile Strength	E 8
Grain Size	E 2
Expansion (Pin Test)	B 153

[a] These designations refer to the following methods of testing:
Tentative Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8),
Tentative Methods of Preparation of Micrographs of Metals and Alloys (A.S.T.M. Designation: E 2), and
Tentative Method of Test for Expansion (Pin Test) of Copper and Copper-Alloy Tubing (A.S.T.M. Designation: B 153).

Tentative Specifications for Copper-Base Alloy Forging Rods, Bars, and Shapes (B 124 – 41 T):

Table I.—Change the chemical composition requirements for alloys Nos. 2 and 11 from their present form to read as follows:

	Alloy No. 2	Alloy No. 11
Copper, per cent	58.00 to 62.00	78.00 to 93.00
Lead, per cent	1.50 to 2.50	....
Tin, max., per cent		0.60
Iron, max., per cent	0.30	4.00
Silicon, max., per cent	....	2.25[a]
Aluminum, per cent	....	6.50 to 11.00
Manganese, max., per cent	....	2.00
Nickel, max., per cent	....	5.50[a]
Zinc, per cent	remainder	.....
Sum of named elements, min., per cent	99.50	99.50

[a] When nickel is present in the alloy, the silicon content shall not exceed 0.25 per cent; conversely, when silicon is present in the alloy, the nickel content shall not exceed 0.25 per cent.

Tentative Specifications for Copper Rods, Bars, and Shapes (B 133 – 40 T):

Section 6.—Change the Rockwell hardness requirements for the hard temper to read as follows by the addition of the italicized figures and the omission of those in brackets:

Temper	Rockwell Hardness			
	B Scale		F Scale	
	Min.	Max.	Min.	Max.
Hard	20	[49] *67*	68	[85] *95*

Tentative Specifications for Brass Wire (B 134 – 41 T):

Section 3.—In order to permit the use of electrolytic cathode copper as an alternate material add a reference to the Tentative Specifications for Electrolytic Cathode Copper (A.S.T.M. Designation: B 115) in the table in Paragraph (*a*) of this section.

Tentative Specifications for Miscellaneous Brass Tubes (B 135 – 40 T):

Section 10.—Change the description of the mercurous nitrate test to read as follows by the addition of the italicized words and the omission of those in brackets:

10. When specifically required, test specimens 6 in. in length of both annealed and drawn tempers shall, *after proper cleaning,* withstand [without cracking] an immersion for 15 min. in an aqueous [mercurous nitrate solution containing] *solution of mercurous nitrate without cracking. Immediately after removal from the solution, the specimen shall be wiped free of excess mercury and examined for cracks. The mercurous nitrate solution shall contain* 100 g. of [mercurous nitrate] $HgNO_3$ and 13 ml. of HNO_3 (sp. gr. 1.42) per liter of solution.

Section 11.—Change from its present form to read as follows:

11. The properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

Test	A.S.T.M. Designation[a]
Tensile Strength	E 8
Rockwell Hardness	E 18
Grain Size	E 2
Expansion (Pin Test)	B 153
Mercurous Nitrate Test	B 154

Tentative Specifications for Phosphor Bronze Rods, Bars, and Shapes (B 139 – 41 T):

Section 3.—Make the same change in these specifications as is recommended above in Section 3 of Specifications B 134.

Tentative Specifications for Tin-Bronze and Leaded Tin-Bronze Sand Castings (B 143 – 41 T):

Table II.—Change the chemical composition requirements for alloy 2A to read as follows by the addition of the italicized figures and the omission of those in brackets:

	Alloy 2A	
	Min.	Max.
Copper, per cent	[86.00] *86.50*	[89.00]
Tin, per cent	5.50	6.50
Lead, per cent	1.00	2.00
Zinc, per cent	2.50	5.00
Nickel, per cent	....	1.00
Iron, per cent	....	[0.20] *0.25*
Phosphorus, per cent.	....	[0.03] *0.05*
Total other constituents, per cent	....	0.35

Table III.—Change the requirement for elongation in 2 in. for alloy 2A from "20" to read "22" min., per cent.

Tentative Specifications for High-Leaded Tin-Bronze Sand Castings (B 144 – 41 T):

New Section.—Add a new Section 15 on certification to read as follows, renumbering the subsequent section accordingly:

15. *Certification.*—In the case of a product manufactured in advance and supplied for sale from stock by the manufacturer, jobber, or other dealer, the product may upon request of the purchaser be certified by the manufacturer as conforming to these specifications subject to the following procedure: Not less than two tension tests, from different heats, and not less than one chemical analysis shall be made by

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[a] These designations refer to the following methods of testing:
Tentative Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8),
Standard Method of Test for Rockwell Hardness of Metallic Materials (A.S.T.M. Designation: E 18 – 36) and
Tentative Method of Test for Rockwell Superficial Hardness of Metallic Materials (A.S.T.M. Designation: E 18 – 39 T),
Tentative Methods of Preparation of Micrographs of Metals and Alloys (A.S.T.M. Designation: E 2),
Tentative Method of Test for Expansion (Pin Test) of Copper and Copper-Alloy Tubing (A.S.T.M. Designation: B 153), and
Tentative Method of Mercurous Nitrate Test for Copper and Copper Alloys (A.S.T.M. Designation: B 154).

the manufacturer from each day's melt. Records of the tension test results and chemical analysis shall be systematically made and maintained and shall be the basis for certification. In lieu of the manufacturer's certification and upon written request by the purchaser, these records may be examined by the purchaser or his accredited representative upon the manufacturer's premises.

Tentative Specifications for Leaded Red Brass and Leaded Semi Red Brass Sand Castings (B 145 – 41 T):

The committee recommends that alloy 5A of these specifications be revised as indicated below. The revised compositions and tensile requirements correspond exactly to those of alloy 5X which has been published in the Emergency Alternate Provisions EA – B 145[2] for use in place of present alloys 4A, 4B, and 5A.

Table II.—Change the chemical composition requirements for alloy 5A to read as follows by the addition of the italicized figures and the omission of those in brackets:

Copper, per cent	[79.00 to 81.75] *78.00 to 82.00*
Tin, per cent	2.50 to 3.25
Lead, per cent	[6.25 to 7.25] *6.00 to 8.00*
Zinc, per cent	[8.00] *7.00* to 10.00
Nickel, max., per cent	1.00
Iron, max., per cent	0.40
Phosphorus, max., per cent	0.02
Total other constituents, max., per cent	0.50

Table III.—Change the tensile requirements of alloy 5A to read as follows by the addition of the italicized figures and the omission of those in brackets:

Tensile strength, min., psi.	[28 000] *29 000*
Yield strength, min., psi.	[12 000] *13 000*
Elongation in 2 in., min., per cent	[15] *18*

Tentative Specifications for Leaded Yellow Brass Sand Castings for General Purposes (B 146 – 41 T):

New Section.—Add a new Section 15 on certification to read the same as new Section 15 recommended above for inclusion in Specifications B 144 – 41 T.

Tentative Specifications for High-Strength Yellow Brass and High-Strength Leaded Yellow Brass Sand Castings (B 147 – 41 T):

New Section.—Add a new Section 14 on certification to read the same as new Section 15 recommended above for inclusion in Specifications B 144 – 41 T.

Tentative Specifications for Aluminum-Bronze Sand Castings (B 148 – 41 T):

Tables I and II.—Omit the requirements for alloy 9A from both of these tables and add the nominal composition and chemical composition requirements for new alloys 9A-1 and 9A-2 in the respective tables to read the same as new alloys 9A-1 and 9A-2 recommended above for inclusion in Tables I and II of Specifications B 30 – 41 T.

New Section.—Add a new Section 14 on certification to read the same as new Section 15 recommended above for inclusion in Specifications B 144 – 41 T.

Tentative Specifications for Leaded Nickel-Brass (Leaded Nickel-Silver) and Leaded Nickel-Bronze (Leaded Nickel-Silver) Sand Castings (B 149 – 41 T):

New Section.—Add a new Section 15 on certification to read the same as new Section 15 recommended above for inclusion in Specifications B 144 – 41 T.

Tentative Specifications for Copper-Nickel-Zinc Alloy Rod and Wire (B 151 – 41 T):

Section 3.—Make the same change in these specifications as is recommended above in Section 3 of Specifications B 134.

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[2] Issued April 6, 1942; printed as a sticker for attachment to Tentative Specifications B 145 – 41 T.

Tentative Specifications for Phosphor Bronze Wire (B 159 – 41 T):

Section 3.—Make the same change in these specifications as is recommended above in Section 3 of Specifications B 134.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption, revisions in certain specifications, as indicated below, and accordingly asks for a nine-tenths affirmative vote at the annual meeting in order that these modifications may be referred to letter ballot of the Society.

Standard Specifications for Copper Bars for Locomotive Staybolts (B 12 – 33):

New Section.—Add a new Section 8 to read as follows to provide for the inclusion of the methods of tension testing in these specifications:

> 8. *Methods of Testing.*—The tensile strength shall be determined in accordance with the Tentative Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8) of the American Society for Testing Materials.

Standard Specifications for Free-Cutting Brass Rod for Use in Screw Machines (B 16 – 41):

Section 4.—In the table of chemical composition requirements, change the fourth item to read as follows by the addition of the italicized word:

> Materials other than copper, lead, *iron*, and zinc, max., per cent.................0.50

New Section.—Add a new Section 8 to read as follows to provide for the inclusion of the methods of tension testing in these specifications:

> 8. *Methods of Testing.*—The tensile strength shall be determined in accordance with the Tentative Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8) of the American Society for Testing Materials.

Standard Specifications for Brass Pipe, Standard Sizes (B 43 – 41):

Section 8.—Change from its present form to read as follows:

> 8. *Mercurous Nitrate Test.*—A test specimen 6 in. in length shall be taken from each pipe selected for test and shall, after proper cleaning, withstand an immersion for 15 min. in an aqueous solution of mercurous nitrate without cracking. Immediately after removal from the solution, the specimen shall be wiped free of excess mercury and examined for cracks. The mercurous nitrate solution shall contain 100 g. of $HgNO_3$ and 13 ml. of HNO_3 (sp. gr. 1.42) per liter of solution.

New Section.—Add a new Section 11 on Methods of Testing to read as follows, renumbering the subsequent sections accordingly:

> 11. *Methods of Testing.*—The properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

Test	A.S.T.M. Designation[a]
Expansion (Pin Test)............	B 153
Mercurous Nitrate Test..........	B 154

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[a] These designations refer to the following methods of testing:
Tentative Method of Test for Expansion (Pin Test) of Copper and Copper-Alloy Tubing (A.S.T.M. Designation: B 153), and
Tentative Method of Test for Mercurous Nitrate Test for Copper and Copper Alloys (A.S.T.M. Designation: B 154).

Standard Specifications for Steam or Valve Bronze Castings (B 61 – 41):

Section 4.—Change the chemical composition requirements to read as follows:

	Min.	Max.
Copper, per cent........	86.50	....
Tin, per cent...........	5.50	6.50
Lead, per cent..........	1.00	2.00
Zinc, per cent..........	2.50	5.00
Nickel, per cent........	....	1.00
Iron, per cent..........	....	0.25
Phosphorus, per cent.....	....	0.05
Total other constituents, per cent.............	....	0.35

TABLE I.—TENSION AND BEND TEST REQUIREMENTS.

Temper	Diameter or Distance Between Parallel Faces	Tensile Strength, min., psi.	Yield [Point] *Strength,*[a] min., psi.	Elongation in 2 in., min., per cent	Bend Test, angle of bend, deg.
TYPES A, C, AND D ALLOY RODS [BARS] AND SHAPES					
Soft	All forms, all sizes	52 000	*15 000*	45	180
Quarter-hard	All forms, all sizes	55 000	24 000	30	120
Half-hard	Rods:				
	Up to ½ in., incl.	70 000	38 000	20	120
	Over ½ to 1 in., incl	70 000	38 000	25	120
	Over 1 to 2 in., incl	70 000	38 000	30	120
	[Bars and] Shapes	[b]	[b]	[b]	[b]
Hard	Rods:				
	Up to ½ in., incl	85 000	50 000	14	90
	Over ½ to 1 in., incl	85 000	50 000	18	90
	Over 1 to 1½ in., incl	85 000	50 000	22	90
	[Bars and] Shapes	[b]	[b]	[b]	[b]
Extra-hard[c]	Rods:				
	Up to ½ in., incl	100 000	55 000	7	60
	[Over ½ to 1 in., incl	100 000	55 000	10	60]
	[Over 1 to 1¼ in., incl	100 000	55 000	14	60]
Types A, C, and D Alloy Bars[d]					
Soft	*All sizes*	*52 000*	....	*45*	....
Hard	*Up to 1 in., incl*	*65 000*	....	*25*	....
	Over 1 to 1½ in., incl	*60 000*	....	*25*	....
	Over 1½ to 3 in., incl	*50 000*	....	*25*	
TYPE B ALLOY RODS, BARS, AND SHAPES					
Soft	All forms, all sizes	40 000	*12 000*	35	180
Half-hard	Rods:				
	Up to ½ in., incl	55 000	20 000	11	180
	Over ½ to 1 in., incl	55 000	20 000	14	180
	Over 1 to 2 in., incl	55 000	20 000	17	180
	Bars and Shapes	[b]	[b]	[b]	[b]
Hard	Rods:				
	Up to ½ in., incl	65 000	35 000	[6] *8*	120
	Over ½ to 1 in., incl	65 000	35 000	[8] *10*	120
	Over 1 to 2 in., incl	65 000	35 000	[10] *12*	120
	Bars and Shapes	[b]	[b]	[b]	[b]
Extra-hard[c] *Bolt temper*	Rods:	[75 000]			
	Up to ½ in., incl	*85 000*	[40 000] *55 000*	[4] *6*	90
	Over ½ to 1 in., incl	75 000	[40 000] *45 000*	[6] *8*	90
	Over 1 to 1½ in., incl	75 000	40 000	[8] *10*	90

---

[a] Stress corresponding to 0.5 per cent strain.

[b] Physical requirements of *types A, C, and D alloy shapes, and type B alloy bars and shapes* of the half-hard and hard tempers as agreed upon between the manufacturer and the purchaser at time of order.

[c] Bars and shapes are not produced in the extra-hard temper.

[d] *Rectangular sections having thicknesses less than the width.*

Section 6.—Change the words "yield point" to read "yield strength."

Standard Specification for Copper-Silicon Alloy Rods, Bars, and Shapes (B 98 – 41):

Table II.—Change to read as shown in the accompanying Table I by the addition of the italicized words and figures and the omission of those in brackets.

New Section.—Add as a new Section 9 a reference to the methods of tension testing, as follows:

> 9. *Methods of Testing.*—The tensile strength shall be determined in accordance with the Tentative Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8) of the American Society for Testing Materials.

Standard Specifications for Copper-Silicon Alloy Wire for General Purposes (B 99 – 41):

Table II.—For type B alloy wire, change the tensile strength requirements for the annealed temper for all diameters from "43,000 to 50,000" to read "43,000 to 55,000" psi., and for the eighth-hard temper, from "50,000 to 60,000" to read "50,000 to 65,000" psi.

New Section.—Add as a new Section 9 a reference to the methods of tension testing to read the same as new Section 9 recommended above for addition to Specifications B 98 – 41.

Standard Specifications for Phosphor Bronze Sheet and Strip (B 103 – 40):

Table I.—Change the requirement for tin in grade A phosphor bronze from "3.80 to 5.80" to read "3.50 to 5.80" per cent.

ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the following two tentative specifications be approved without revision for reference to the letter ballot of the Society for adoption as standard:

Tentative Specifications for Copper-Silicon Alloy Plate and Sheet for Pressure Vessels (B 96 – 41 T), and

Tentative Specifications for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 – 40 T).

ADOPTION OF TENTATIVE REVISION OF STANDARD

The committee recommends the adoption of the tentative revision issued in 1941 in the Standard Specifications for Phosphor Bronze Sheet and Strip (B 103 – 40).

WITHDRAWAL OF STANDARD

The committee recommends that the Standard Specifications for Muntz Metal Condenser Tube Plates (B 57 – 27) be withdrawn, since they have been replaced by the new Tentative Specifications for Copper-Alloy Condenser Tube Plates (B 171 – 42 T) which were accepted for publication as tentative by Committee E-10 on Standards, March 27, 1942, and have been issued as a separate reprint.

APPENDIX II

STUDIES OF MACHINED *versus* AS-CAST TEST SPECIMENS FOR COPPER-BASE ALLOY SAND CASTINGS

Report to Subcommittee X on Copper-Base Alloys for Sand Castings
by J. J. Curran,[1] J. W. Bolton,[2] F. L. Wolf,[3] and W. E. Martin[4]

TENSION TEST BARS FOR BRASS AND BRONZE CASTINGS

By J. J. Curran[1]

Standard specifications vary considerably with respect to requirements for specimens for the tension test. Specifications for wrought steels specify dimensions of the specimen to be used and how it shall be cut from the steel shape. In the case of cast steels, the dimensions to which the specimen is to be machined are usually specified, and it is assumed that it will be machined from a specially cast test coupon, but it is not customary to specify closely how this coupon shall be cast.

In the case of cast iron, the specimen is sometimes cast to size and shape, in accordance with specific directions, and is tested without machining the test length. Some specifications require the test specimen to be machined completely from a cast transverse test bar. In the case of malleable iron, the specimen is cast to size and tested without machining.

A.S.T.M. specifications for most copper-tin-zinc and copper-tin-lead-zinc bronzes specify not only that the test specimen shall be machined all over, but likewise indicate how the coupon shall be cast, that is, they specify the actual dimensions of the coupon and feeding head. This practice is also followed in Navy and Federal specifications, where even the location of the gate is specified.

It is our opinion, and we will attempt to substantiate it, that the present A.S.T.M., Navy, and Federal test coupons are not ideal; therefore, we favor an investigation leading to the adoption of a new coupon which will approximate more closely the ideal, and perhaps also which may be tested without machining or may be machined at the option of the producer. Due to the relatively small size of average heats of bronze, the cost of a machined test specimen from each heat is prohibitive. A specimen which could be tested without machining would be considerably less expensive and hence would permit better coverage of the metal cast each day, as well as eliminating the delay incident to machining.

For some years A.S.T.M. specifications for brass and bronze castings have required that the test specimen be machined to size from a coupon cast to shape, with a wedge-shaped feeding head joined by a web to the coupon proper and running its entire length. The coupon so cast is commonly referred to as the Webbert test bar.

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[1] Chief Metallurgist, Walworth Co., Inc., Greensburg, Pa.

[2] Director of Metallurgical Research and Testing, The Lunkenheimer Co., Cincinnati, Ohio.

[3] Chief, Graphite Allocation and Conservation, Materials Division, Branch K, War Production Board, Washington, D. C.

[4] Materials Laboratory, Sperry Gyroscope Co., Brooklyn, N. Y.

Navy and Federal specifications differ from those of the A.S.T.M. in that they specify not only most of the important dimensions, but also indicate that the pattern is to be gated to the bottom of the feeding head, just above the web, rather than to the coupon proper. A.S.T.M. specifications omit all reference to the location of the gate. Navy and Federal specifications likewise permit the use of an optional coupon, practically identical with the Webbert test bar, except that the feeding head does not contact the test length of the coupon. This modified Webbert coupon is known as the Crown coupon.

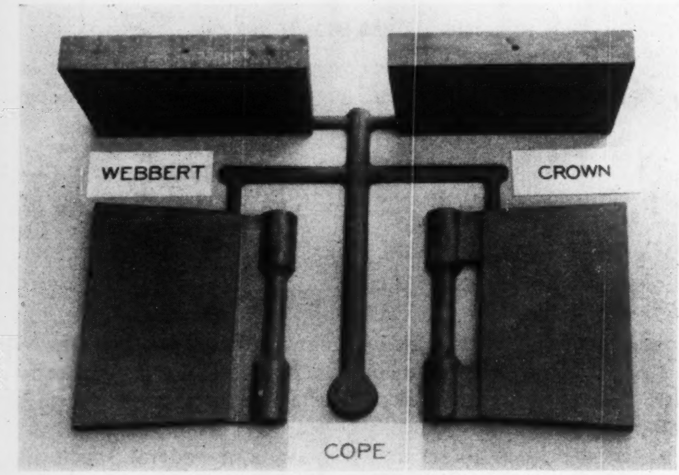

FIG. 1.—Arrangement of Patterns and Gating for Casting Webbert and Crown Bars with Feeding Head Vertical in Cope.

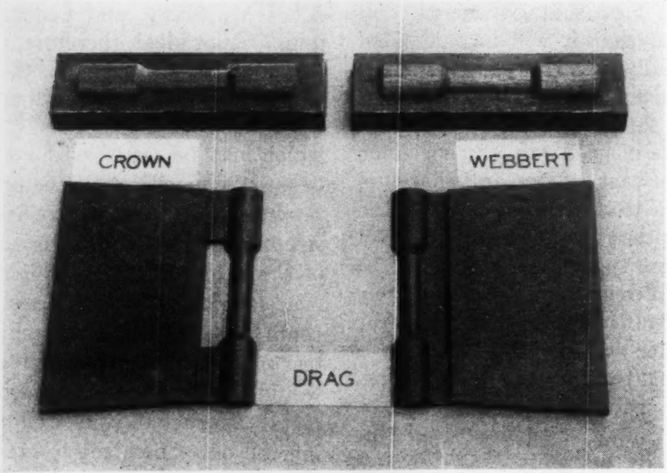

FIG. 2.—Arrangement of Patterns for Casting Webbert and Crown Bars with Feeding Head Horizontal, Half in Cope and Half in Drag.

When a test coupon is thus carefully specified as to dimensions, feeding, and location of gate, it is reasonable to assume that the design is the result of long experience and careful planning. It is a fact, however, that test bars machined from either the Webbert or Crown coupon frequently tend to break in the fillet or shoulder, giving low values for elongation and sometimes also for tensile strength. The reason for shoulder breaks is conjectural, but it does indicate that the carefully specified coupon and feeding head are not ideal by a great margin.

The logical method of casting either the Webbert or Crown coupon is with the feeding head in the vertical position, above the coupon proper. With the gate located as specified by Navy and Federal specifications it is necessary practically that the coupon and web be entirely in the drag of the mold with the feeding head in the cope, using a dry sand core in the drag to shape the web and top half of the coupon.

An alternative method is to mold the pattern horizontal on the parting line, half in the drag and half in the cope, and then turn the mold on edge for pouring, in order to have the head over the coupon.

These methods make molding of test bars somewhat complicated, and some foundries have resorted to the practice of casting the coupon with the web and feeding head horizontal on the parting line, half in the drag and half in the cope. Whereas this practice seems illogical, it does, strangely enough, frequently give better tensile properties than when the feeding head is where it should be, above the coupon. Even when cast in this manner, however, shoulder breaks are quite frequent.

We have heard with interest the claims for superiority of one type of coupon over the other, and we have run some tests in the past which seemed to indicate that when cast with the feeding head vertical, the best and most consistent results were obtained with the Crown type coupon. However, we have always wondered at the reasons for the superiority of the results from the illogical horizontal method of casting. We have had to conclude that something is basically wrong with the design of the coupon and feeding head.

In order to develop some data on the question, a plated pattern was made by which a Webbert and a Crown coupon could be cast in the drag with the heads vertical above them, using dry sand cores to form the webs and upper halves of the test bars. On the same plate were mounted patterns for both types of bars with the heads horizontal, that is, half in the cope and half in the drag. All four patterns were gated at the point specified in the Navy specifications and were connected by small runners to a common sprue. The arrangement of the patterns and the gating employed are shown in Figs. 1 and 2. The sprue itself is not shown, as this was cut by hand during molding.

Test bars were cast from this pattern, using ten heats of valve bronze conforming to A.S.T.M. Standard Specifications for Steam or Valve Bronze Castings (B 61 – 41)[5] and Navy Department Specification 46B8g for Bronze, Valve (Composition M) Castings. The metal was melted in commercial units for valve and fittings manufacture, from uniform charges of ingot and foundry returns. The pouring temperatures of the heats were varied to determine if pouring temperature was an important factor in the behavior of the different types of test bars molded in the two different positions. The chemical compositions of the test bars as determined by analysis are shown in Table I.

After being machined to size the specimens were broken in the tension test. The tensile properties observed are shown in Table II, in which the heats are arranged in ascending order of pouring temperatures. All four bars from each heat are grouped together. The marks W-V, W-H, C-V, and C-H in the third column indicate the type of bar and the method of molding as explained in the footnote to the table.

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[5] 1941 Supplement to Book of A.S.T.M. Standards, Part I, pp. 159, 162.

It is unfortunate that only one coupon of each type and each method of molding was available in this series of tests. Due to the fact that a second test bar was not available in cases of shoulder breaks and flaws, some abnormally low values are reported. Normally, a duplicate bar could be broken with the chance that these values would be improved considerably.

TABLE I.—CHEMICAL COMPOSITION OF HEATS USED IN TEST BAR INVESTIGATION.

Heat	Copper, per cent	Tin, per cent	Lead, per cent	Zinc, per cent	Nickel, per cent	Iron, per cent
1	88.04	6.20	1.64	3.35	0.70	0.06
2	87.70	6.26	1.62	3.62	0.68	0.07
3	87.49	6.26	1.76	3.70	0.71	0.07
4	87.95	6.30	1.57	3.40	0.70	0.06
5	87.62	6.00	1.84	3.72	0.74	0.07
6	87.72	6.20	1.66	3.65	0.70	0.06
7	87.65	6.23	1.70	3.65	0.70	0.06
8	87.50	6.12	1.89	3.68	0.73	0.07
9	87.63	6.28	1.59	3.70	0.72	0.07
10	87.82	6.26	1.64	3.51	0.70	0.06

In Fig. 3 are compared the tensile strength values for the Webbert and Crown bars cast by both vertical and horizontal methods. In Fig. 4 the elongation values are similarly compared.

Conclusions:

Study of the tension test results for this series of test bars discloses the following:

1. Eight out of thirty-eight test specimens broke in the fillet, outside the gage marks. Only one of these shoulder breaks occurred in a Crown type bar.

2. The greater number of shoulder breaks occurred at low pouring temperatures, though this might not follow in a more comprehensive series of tests.

TABLE II.—RESULTS OF TENSION TESTS.

Heat	Poured	Test Bar[a]	Tensile Strength, psi	Elongation in 2 in., per cent
1	2030 F.	W-V	43 500	33.0
		W-H	46 000	36.0[b]
		C-V	42 100	37.0
		C-H	[c]	[c]
2	2060 F.	W-V	37 000	20.0[b]
		W-H	42 200	26.0[b]
		C-V	43 500	36.0
		C-H	27 600	6.5[d]
3	2090 F.	W-V	40 900	43.5
		W-H	38 750	19.5[b]
		C-V	36 500	17.0[b]
		C-H	37 400	25.0
4	2110 F.	W-V	40 850	30.0
		W-H	43 500	35.5
		C-V	40 000	27.0
		C-H	39 000	28.0
5	2140 F.	W-V	33 250	15.5[b]
		W-H	38 400	25.0
		C-V	38 500	27.0
		C-H	37 500	24.0
6	2150 F.	W-V	36 600	23.0
		W-H	40 500	40.0
		C-V	38 000	26.0
		C-H	36 000	21.0
7	2170 F.	W-V	38 700	25.0
		W-H	38 600	25.0[b]
		C-V	33 600	27.0
		C-H	33 500	19.5
8	2190 F.	W-V	34 000	22.5
		W-H	38 200	25.0
		C-V	36 500	22.5
		C-H	35 750	22.5
9	2210 F.	W-V	38 350	30.0
		W-H	38 000	21.0[b]
		C-V	[e]	[e]
		C-H	38 000	25.5
10	2220 F.	W-V	36 850	27.0
		W-H	41 700	40.0
		C-V	39 500	28.0
		C-H	41 400	40.0

[a] W-V, a Webbert bar molded with feeding head vertical in cope.

W-H, a Webbert bar molded with feeding head horizontal, half in cope and half in drag.

C-V, a Crown bar molded with feeding head vertical in cope.

C-H, a Crown bar molded with feeding head horizontal, half in cope and half in drag.

[b] Broke in fillet, outside gage marks.

[c] Metal cold, did not run.

[d] Flaw.

[e] Bar damaged in machining.

3. The method of casting, that is, vertical *versus* horizontal, does not appear to influence the frequency of shoulder breaks.

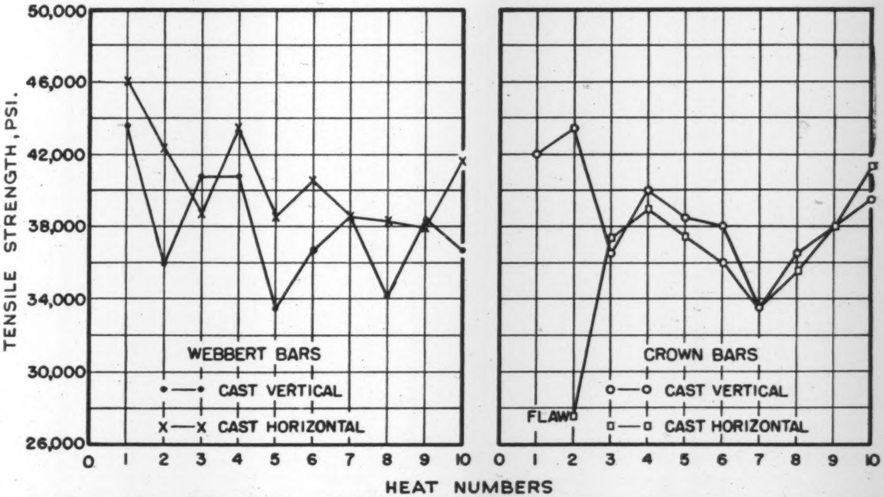


FIG. 3.—Comparison of Tensile Strength Values for Webbert and Crown Bars Cast by Both Vertical and Horizontal Methods.

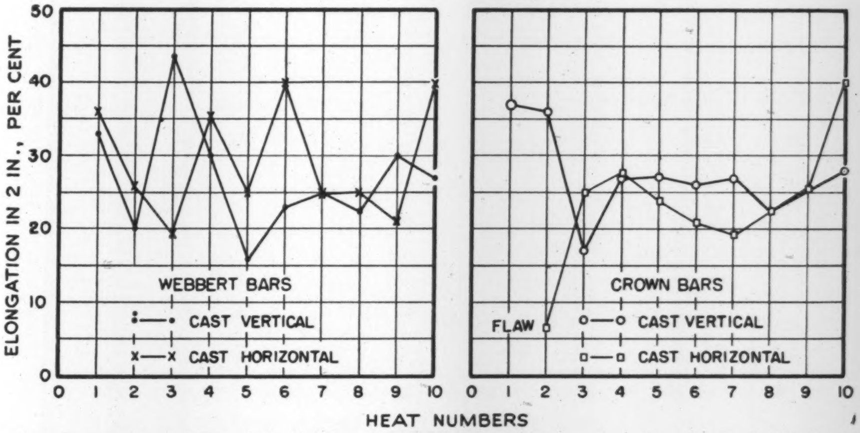


FIG. 4.—Comparison of Elongation Values for Webbert and Crown Bars Cast by Both Vertical and Horizontal Methods.

4. There is no evidence to indicate that shoulder breaks are due to faulty machining or improper testing, hence, are probably due to weakness of the metal in the fillet area.

5. From heat to heat, wider variations in tensile strength and elongation are encountered in the Webbert bars than in the Crown bars.

6. Webbert bars cast horizontally give generally substantially higher test values than those cast vertically. The method of casting seems to have less effect on the Crown bars.

From these results it is evident that this type of bar, either Webbert or Crown, is subject to weakness in the fillet area. This weakness tends to give fictitious values for tensile strength and elongation. The cause of this weakness is probably improper feeding of the test bar itself. This conclusion is supported by the fact that by casting horizontally, so that the feeding head has little or no opportunity to function, equally good or better results can be obtained.

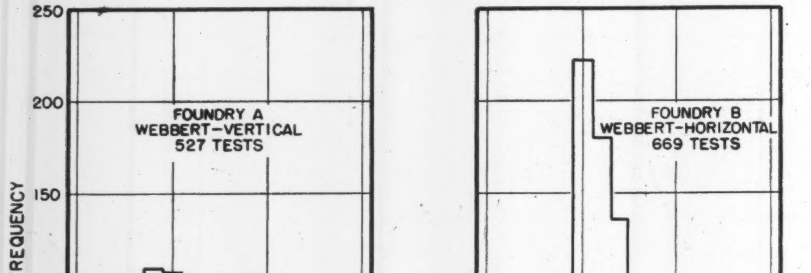


FIG. 5.—Frequency Curves for Tensile Strengths of Webbert Bars Cast by Both Vertical and Horizontal Methods.

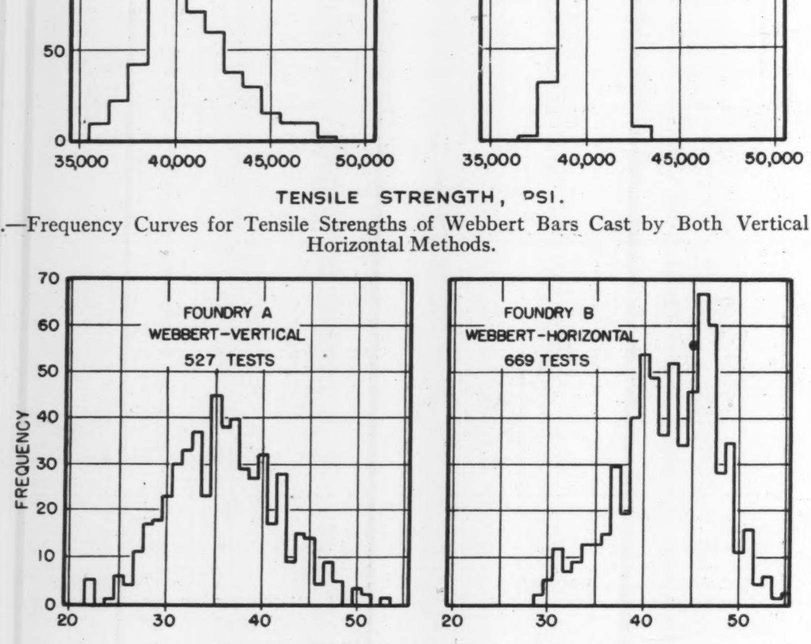


FIG. 6.—Frequency Curves for Elongations of Webbert Bars Cast by Both Vertical and Horizontal Methods.

A statistical study was made of the tensile properties obtained on this same metal in two different foundries, both of which used the same foundry methods for producing the same type of castings. One foundry, however, used the Webbert coupon with the head vertical above it, and the other cast the coupon with the head horizontal on the parting line.

The data for this study were from a

periods elected at random, and represent all of the control tests made on this particular material in each foundry during that period.

The frequency curves for tensile strength in Fig. 5 indicate a slight superiority in favor of the horizontal method of casting. In Fig. 6 it is evident the elongation is improved substantially by casting the coupon with the head horizontal. These frequency curves corroborate statistically the indications of the experimental heats previously described.

The basic purpose in specifying minimum tensile properties in standard specifications for cast metals is to insure that the metal at the instant of pouring is of good quality, and has nothing whatsoever to do with the properties which may be expected in any and all sections of any and all castings poured from that metal.

The test coupon, therefore, should be a practically ideal casting, ideally gated and fed to eliminate the variables which produce the variations in properties known to exist in different sections of intricate castings. The test specimen should show the metal at its best, free from the prejudicial effects of inadequate design.

The tests reported here indicate that the present A.S.T.M. coupon for bronzes does not constitute an approximately ideal casting. It would seem in order, therefore, that Subcommittee X, of Committee B-5, give consideration to a new design of coupon which will reflect accurately and consistently the true quality of the metal at the time of pouring into the mold. Further, consideration should be given to the problem of a test coupon which can be tested in the as-cast condition, as a means of economy of time and labor. If possible, the coupon should be one that could be tested as cast or after machining, at the option of the producer.

COMPARISON OF A.S.T.M. AND OTHER TEST SPECIMENS FOR BRONZE CASTINGS

BY J. W. BOLTON[2]

This report presents data showing comparisons of the prescribed A.S.T.M. test specimen for bronze castings (referred to as the A.S.T.M. specimen, Fig. 7) and a control test bar of The Lunkenheimer Co. (referred to as LTB-1 bar, Fig. 8). A majority of comparisons are presented on the alloy known as steam bronze and Navy M metal covered in the A.S.T.M. Standard Specifications for Steam or Valve Bronze Castings (B 61–41)[5] and the Federal Specification for Bronze Castings (QQ-B-691a). Some comparisons are also given on the 85–5–5–5 alloy covered by the A.S.T.M. Standard Specifications for Composition Brass or Ounce Metal Castings (B 62-41),[5] and also on a silicon brass. Some other types of bars are also considered.

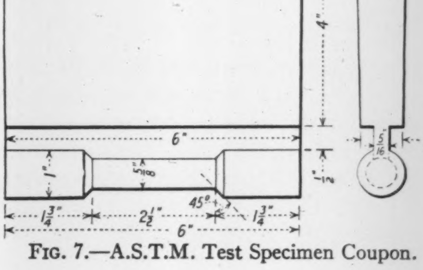
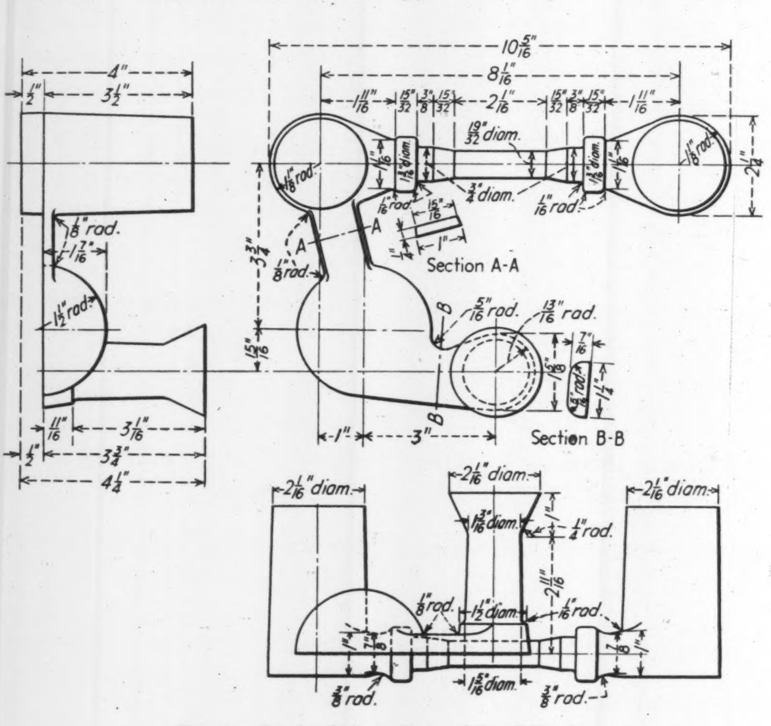


FIG. 7.—A.S.T.M. Test Specimen Coupon.

In the comparisons described herein care was taken to have all conditions as alike as possible, the major variable then being the gating of the coupon.

Metal in each case was poured from the same ladle into different design coupon molds. These molds, made from the same green sand, were placed side by side for pouring. Coupons were not removed from the molds until 3 hr. or more had elapsed.



4"
½"
3½"
⅛" rad.
1 7/16
1½" rad.
11/16
3 1/16
½"
3¾"
4¼"
10 5/16"
8 1/16"
1 11/16"
15/32"
3/8"
15/32
2 1/16"
15/32"
3/8"
15/32"
1 11/16"
19/32" diam.
1 1/16
1 3/16" diam.
1 1/16"
¾" diam.
1/16" rad.
1/16" rad.
1⅛" rad.
1⅛" rad.
2¼"
15/16"
1"
¼"
Section A-A
A
A
⅛" rad.
3¾"
15/16"
B
5/16" rad.
13/16" rad.
7/16"
1 5/16"
1¼"
Section B-B
B
1"
3"
2 1/16" diam.
2 1/16" diam.
2 1/16" diam.
1 3/16" diam.
1"
¼" rad.
2 11/16"
⅛" rad.
1½" diam.
1/16" rad.
1"
7/8
7/8
1"
1 5/16" diam.
3/8" rad.
3/8" rad.

FIG. 8.—Lunkenheimer Control Bar LTB-1.

The metal used was of regular commercial quality, from 900-lb. melts, and was melted with Orsat gas analysis and proportioning burner control of atmospheres in furnaces of the Lunkenheimer open flame design. The burners were regulated to give 0.0 to 0.5 per cent excess oxygen in the gases after combustion. An alkaline slag, largely sodium carbonate, was employed. The temperature of the melt in the furnace was controlled by pyrometers. The metal was poured from the furnaces into pots or ladles holding about 200 to 250 lb. Pouring temperatures were also controlled pyrometrically.

The B 61 alloy was held rather closely to 6.0 per cent tin. An increase of tin of about 0.3 per cent would result in 1500 psi. higher tensile strengths at room temperature than are shown.

The dimensions of the A.S.T.M. specimen Fig. 7, were maintained. Gating was through the bottom half at the end of the bar.

The Lunkenheimer control bar, LTB-1, is shown in Fig. 8. This is a simple squeezer type casting and the molds can be made very rapidly. The use of ball gate and adequate risers also is standard in our production of critical pressure castings. The test bar can be tested as cast or after machining the test length. The fixture shown in Fig. 9 is suitable for applying load to the specimen without threading the ends for grips. Breaking of the test bar at the shoulders does not occur and breakage at the gage marks is infrequent. Alignment is good.

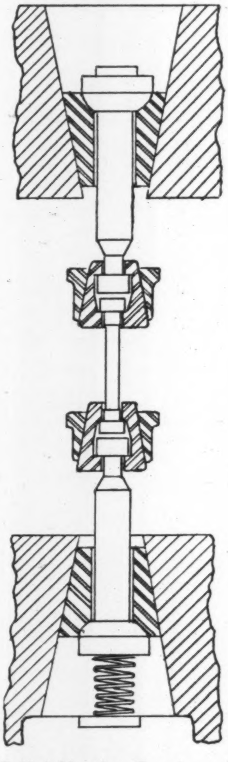


FIG. 9.—Self Adjusting Specimen Holders.

The Federal QQ-M-151a bar, Fig. 10 prescribes ingress gate going into the riser, a not too desirable practice with green sand molds.

Comparative Tests, B 61 Alloy:

Results from some 26 consecutive tests are shown in Figs. 11, 12 and 13. These figures are self explanatory. The Lunkenheimer bar shows higher tensile strength, higher elongation, and higher yield strength, together with greater uniformity in these properties. This is evident in both machined and unmachined LTB-1 bars. It will be noted that the advantages are present at both ranges of pouring temperature with the exception that variation in elongation from heat to heat is somewhat greater at lower pouring temperature. However, in all cases the average and minimum values for all properties are higher for the LTB-1 bar as compared to the A.S.T.M. specimen.

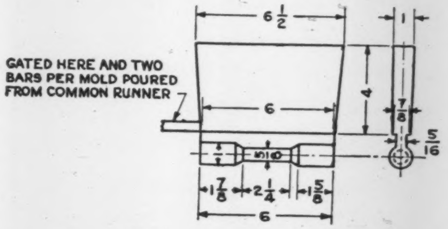
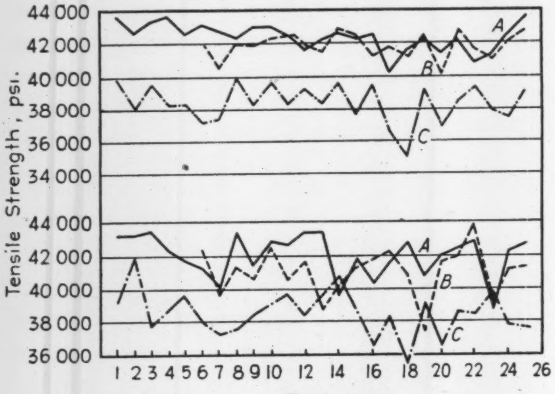


FIG. 10.—Test Bar Prescribed in Fig. 10 of Federal Specification QQ-M-151a.

The unmachined LTB-1 bars average slightly higher in tensile and yield strengths as compared to the machined LTB-1 bars, and slightly lower in ductility. The elongations obtained on the A.S.T.M. specimens are particularly deficient.

With the thought that the dimensions of web and riser of the A.S.T.M. specimen are wrong, and the principle of riser above the casting possibly right, the pattern shown in Fig. 14 (LTB-3 bar) was used. (This pattern is well suited to alloys containing from about 30 to 70 per cent nickel.) The results on



Curve A—LTB-1 bar, unmachined.
Curve B—LTB-1 bar, machined.
Curve C—A.S.T.M. specimen.

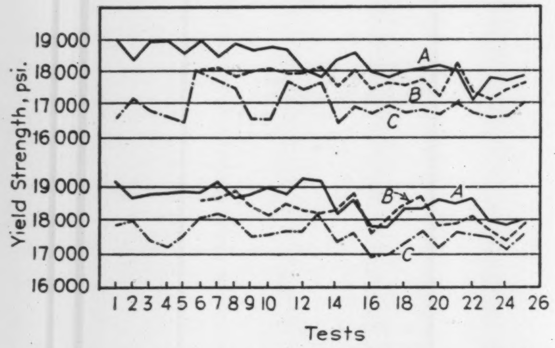
Poured at 2150–2190 F.

	Avg.	Max.	Min.	Variation
A....	42 348	43 600	40 000	3 600
B....	41 830	42 800	40 000	2 800
C....	38 023	40 200	36 300	3 900

Poured at 2050–2100 F.

	Avg.	Max.	Min.	Variation
A....	41 976	43 000	38 400	4 600
B....	41 015	43 900	37 100	6 800
C....	38 503	42 000	35 400	6 600

FIG. 11.—Comparison of Tensile Strength Results on A.S.T.M. B 61 Alloy Determined on LTB-1 Bars Unmachined and Machined and on A.S.T.M. Specimen.



Curve A—LTB-1 bar, unmachined.
Curve B—LTB-1 bar, machined.
Curve C—A.S.T.M. specimen.

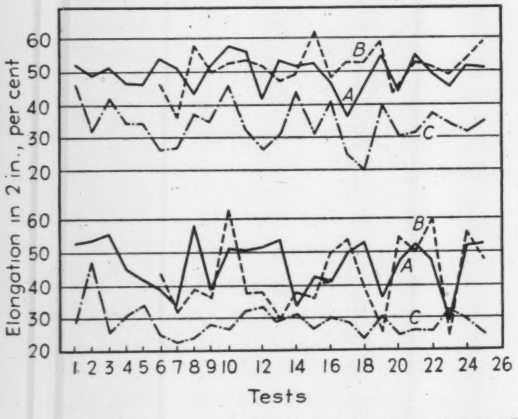
Poured at 2150–2190 F.

	Avg.	Max.	Min.	Variation
A....	18 343	19 000	17 200	1 800
B....	17 820	18 200	17 200	1 000
C....	17 051	18 100	16 500	1 600

Poured at 2050–2100 F.

	Avg.	Max.	Min.	Variation
A....	18 608	19 300	17 800	1 500
B....	18 215	18 900	17 400	1 500
C....	17 597	18 400	16 800	1 600

FIG. 12.—Comparison of Yield Strength Results on A.S.T.M. B 61 Alloy Determined on LTB-1 Bars Unmachined and Machined and on A.S.T.M. Specimen.



Curve A—LTB-1 bar, unmachined.
Curve B—LTB-1 bar, machined.
Curve C—A.S.T.M. specimen.

Poured at 2150–2190 F.

	Avg.	Max.	Min.	Variation
A....	49.7	57.8	35.5	22.3
B....	51.5	61.7	35.4	26.3
C....	33.9	47.4	19.6	27.8

Poured at 2050–2100 F.

	Avg.	Max.	Min.	Variation
A....	46.1	57.8	29.4	28.4
B....	42.7	62.6	25.1	37.5
C....	28.9	47.3	22.3	25.0

FIG. 13.—Comparison of Elongation Results on A.S.T.M. B 61 Alloy Determined on LTB-1 Bars Unmachined and Machined and on A.S.T.M. Specimen.

A.S.T.M. B 61 alloy are shown in Fig. 15, and it will be noted that the LTB-3 bar with its heavy over-all riser shows slightly lower tensile strength and a little greater ductility than the Lunkenheimer control bar LTB-1. The data clearly indicate that the proportions of the riser and the size of the web on the A.S.T.M. coupon are wrong for the bronze considered. However, the LTB-3 bar has many of the disadvantages of high cost shared by the A.S.T.M. specimen.

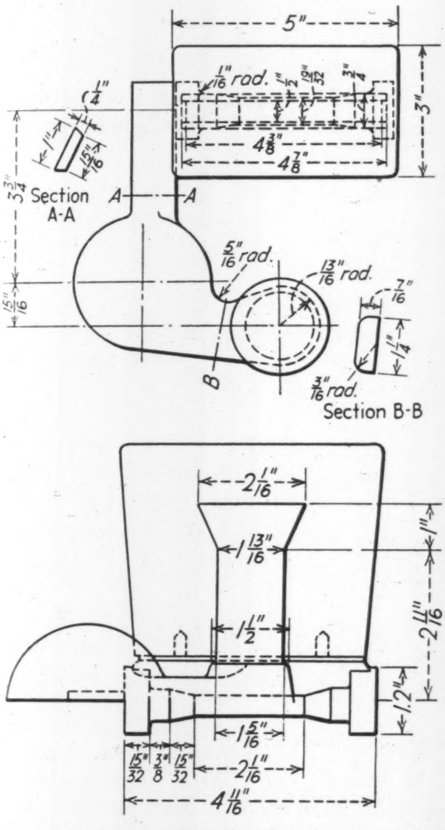


FIG. 14.—Lunkenheimer LTB-3 Bar, Cast with Riser Vertically Above the Casting.

When high shrinkage alloys, such as manganese bronze, nickel alloys, and others, are cast, more liquid shrinkage is involved and an over-all riser often is justified. Even in the case of alloys with shrinkage characteristics slightly higher than tin bronzes, an over-all riser may be desirable. For example, a silicon brass or bronze (3.5 per cent silicon, 13 per cent zinc) showed, in comparison tests, much better results for the A.S.T.M. specimen than for the LTB-1 bar. The results of these tests appear in Table III.

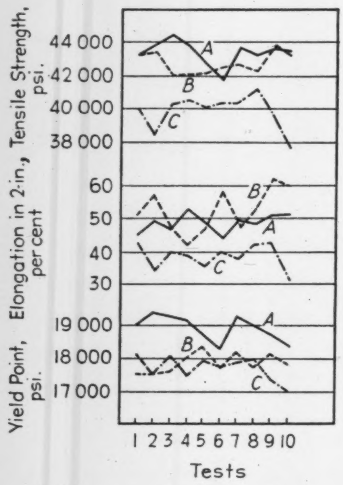
There is a principle that should be followed in all specifications for test specimens, as follows: *Test bar prescriptions should follow sound foundry gating practices. There should be different bar prescriptions for different classes of alloys.*

TABLE III.—RESULTS OF TENSION TESTS ON SILICON BRASS DETERMINED ON A.S.T.M. SPECIMENS AND LTB-1 CONTROL BARS.

Heat	Tensile Strength, psi.		Yield Strength, psi.		Elongation in 2 in., per cent	
	A.S.T.M. Specimen, Fig. 7	LTB-1 Bar, Fig. 8	A.S.T.M. Specimen, Fig. 7	LTB-1 Bar, Fig. 8	A.S.T.M. Specimen, Fig. 7	LTB-1 Bar, Fig. 8
1........	67 300	55 700	26 500	25 300	37.7	18.0
2........	68 400	61 100	26 100	25 500	37.3	25.3
3........	67 100	64 800	27 800	26 200	28.3	24.2
4........	67 600	59 000	26 700	25 300	36.4	20.6
5........	65 800	65 700	27 600	27 300	23.0	27.9
6........	68 500	59 800	26 000	25 500	40.0	24.4
7........	65 700	63 700	26 200	26 000	32.0	29.5
8........	68 700	59 500	26 600	24 500	37.1	22.1
9........	68 700	67 300	27 500	27 000	32.0	31.3
10........	61 100	56 500	25 800	25 900	24.9	18.0
Average	66 890	61 310	26 680	25 850	32.9	24.1

Studies of Routine Control Bars:

There is much to be learned from a study of test bars. The Lunkenheimer Co. consistently works toward promoting the density of castings by the use of deep etch tests, study of microstructure, etc. Studies of the effects of casting practices on test bars indicate in a measurable way the effects of melting, sand, pouring temperatures, etc. Metal of high strength and ductility has the most dense structure, chemical composition being constant. An unreliable or faulty method of making test bars confuses the real issue. With the well gated bar, if strength drops off, foundry practices demand prompt investigation. With unreliable gating one does not know whether trouble is the result of bad metal, or of a faulty test. There is objection to bars which, cast under the same sand condition as the castings they represent, give results known to be low due to bad design and not to bad metal. Nor do good results on any test bar necessarily reflect best properties in the castings—even if the castings do pass pressure tests. The lessons of the test bars must be projected into the production of castings.



Curve A—LTB-1 bar
Curve B—LTB-3 bar
Curve C—A.S.T.M. specimen

		Avg.	Max.	Min.	Variation
Tensile Strength, psi.	A..	43 380	44 300	41 800	2 500
	B..	42 720	43 800	42 000	1 800
	C..	39 840	41 100	37 500	3 600
Elongation in 2 in., per cent	A..	48.86	52.8	44.0	8.8
	B..	52.53	61.7	42.3	19.4
	C..	38.80	43.8	30.4	13.4
Yield Strength, psi.	A..	18 930	19 400	18 300	1 100
	B..	17 920	18 400	17 500	900
	C..	17 630	18 100	17 000	1 100

FIG. 15.—Comparison of Tension Test Results on A.S.T.M. B 61 Alloy Determined on LTB-1 and LTB-3 Test Bars and on A.S.T.M. Specimen.

Study of test bars of exceptionally high strength, such as 48,000 to 51,000 psi. tensile strength, and 55 to 60 per cent elongation, which occur once in a while over many hundreds of tests, suggest that under ideal conditions the potential strength of the B 61 alloy is higher than is attained in usual practice. Careful examination of bars having tensile strengths of 44,000 psi. and above shows a very high degree of density, as seen under the microscope. Conversely, when tensile strengths as low as 38,000 psi. are encountered, microscopic shrinkages can be seen. About 88 per cent of the test results are in the range from 40,000 to 44,500 psi. tensile strength, and 97 per cent have a tensile strength of 38,000 psi. and above, as determined on the control bar. With furnace atmospheres, temperature, and sand under strict technical control (as opposed to guessing at flames, temperatures, and sand conditions), the 3 per cent of the tests which showed tensile strengths of 36,000 up to 37,500 psi. (which always are traced back) usually are due to carelessness in pouring.

With 6 per cent tin in the alloy, a tensile strength of 42,000 to 42,500 psi. is about average. This average is increased about 1000 psi. for each 0.2

per cent additional tin, or say to 44,000 psi. average tensile strength at 6.4 per cent tin. This factor of tin content must be watched when attempting comparisons.

Accelerated Cooling Undesirable:

It was pointed out recently by W. B. George[6] that "foundries who have had the most outstanding results on test bars are those who are using dry sand cores for molds." This quite possibly is so, but somewhat beclouds the real issue. One could go further and cast the metal into metal molds or chills and get high test results. It has been recognized for many years that gassed metal can be disguised by any means of extra rapid cooling. That is just what is *not* wanted in test bars; these bars *should* reflect bad melting practices. In our research studies we have deliberately produced badly gassed metals, and invariably porosity (incipient shrinkage) that shows up in the microstructure is reflected also in low tensile strength and poor ductility. *The test bar should be a measure of the potential soundness of the metal as it goes to the molds.* A poorly gated and fed design, such as the A.S.T.M. coupon Fig. 7, introduces a variable aside from metal quality. Any method which promotes ultra fast cooling also prevents measure of the true quality of the metal.

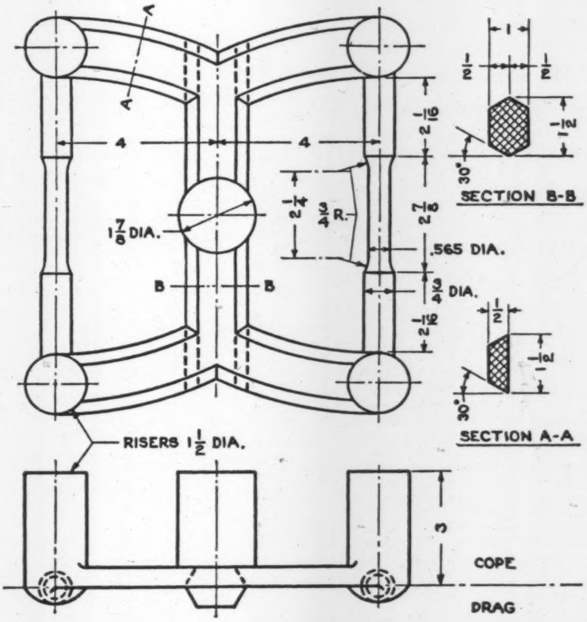


FIG. 16.—Test Bar Prescribed in Fig. 11A of Federal Specification QQ-M-151a.

Results on Federal Test Bars:

The test bar prescribed in Fig. 11a of Federal Specification QQ-M-151a and shown in the accompanying Fig. 16 frequently is used for cast aluminum alloys. This is a cast-to-size bar pulled as cast, or with the gage length skin machined as may be desired. A comparison of the results obtained with A.S.T.M. alloy B 61 using this Federal test bar, Fig. 16, and the LTB-1 bar are shown in Table IV. Tests on the LTB-1 bars average appreciably higher, and give much more uniform results—a spread of 2700 psi. compared with 4500 psi. on the Federal bar shown in Fig. 16. Results on the LTB-1 bar also average much higher in ductility than those on the Federal bar.

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[6] W. B. George, "Some Causes of Porosity Occurring in Navy G and Similar Alloys," *American Foundryman*, Vol. 5, No. 3, March, 1943.

An objection to the test bars prescribed in Figs. 11a and 11b of Federal Specification QQ-M-151a is the long cylindrical ends. Our experience is that the "button" ends, as in the LTB-1 bar, used with a suitable test fixture (as Fig. 9) give better alignment. Long cylindrical ends in V-grips sometimes slip, and misalignment results. Ordinarily, the ends would be threaded before testing, but since the Federal bar shown in Fig. 16 is intended to be pulled without any machining, tests herein reported were not threaded or otherwise machined.

TABLE IV.—RESULTS OF TENSION TESTS ON A.S.T.M. B 61 ALLOY DETERMINED ON LTB-1 CONTROL BARS AND FEDERAL TEST BARS.

Heat	Tensile Strength, psi.		Yield Strength, psi.		Elongation in 2 in., per cent	
	Federal Bar, Fig. 16	LTB-1 Bar, Fig. 8	Federal Bar, Fig. 16	LTB-1 Bar, Fig. 8	Federal Bar, Fig. 16	LTB-1 Bar, Fig. 8
1	39 300	43 000	18 300	18 800	26.9	51.2
2	40 600	41 700	17 700	17 900	33.4	41.6
3	40 100	42 500	18 700	18 600	28.4	47.2
4	43 600	43 000	19 100	18 600	37.6	51.3
5	40 500	41 700	17 900	18 600	33.1	43.4
6	39 200	43 800	18 300	19 100	26.3	52.7
7	39 400	42 800	17 700	18 700	33.3	48.9
8	42 700	42 700	18 300	18 500	43.4	48.1
9	43 000	42 800	18 200	18 600	35.8	45.2
10	42 800	42 100	18 700	18 500	42.3	42.5
11	39 100	43 400	18 500	18 400	27.6	50.7
12	41 100	44 400	17 500	18 800	41.5	55.8
Average	40 953	42 825	18 233	18 591	34.1	48.2

Test on A.S.T.M. B 62 Alloy:

The 85–5–5–5 alloy covered by the A.S.T.M. Standard Specifications for Composition Brass or Ounce Metal Castings (B 62 - 41)[5] can be made to give good strength values as shown by the test results obtained on consecutive heats using the LTB-1 control bar which are reported in Table V.

As is shown in Table VI the better feeding of the Lunkenheimer control bar LTB-1 is productive of higher tensile strength and much better ductility. Noteworthy is the fact that eight out of ten A.S.T.M. bars, Fig. 7, broke beyond the gage marks, usually at the shoulder.

TABLE V.—CHEMICAL COMPOSITION AND RESULTS OF TENSION TESTS ON A.S.T.M. B 62 ALLOY (85–5–5–5) DETERMINED ON LTB-1 CONTROL BARS.

Heat	Chemical Composition			Physical Properties		
	Tin, per cent	Zinc, per cent	Lead, per cent	Yield Strength, psi.	Tensile, Strength, psi.	Elongation in 2 in., per cent
1	4.82	4.59	5.14	17 700	38 700	31.7
2	4.87	4.69	4.80	18 300	40 000	36.8
3	4.58	4.96	4.72	20 100	37 500	29.9
4	4.63	4.95	4.90	17 700	38 100	30.8
5	4.77	3.96	4.59	17 600	37 900	32.3
6	4.65	3.66	5.25	17 400	38 200	28.8
7	4.76	3.84	4.65	18 400	40 800	36.8
8	4.67	4.02	4.40	17 800	38 300	33.3

TABLE VI.—RESULTS OF TENSION TESTS ON A.S.T.M. B 62 ALLOY (85–5–5–5) DETERMINED ON A.S.T.M. SPECIMENS AND LTB-1 CONTROL BARS.

Heat	Tensile Strength, psi.		Elongation in 2 in., per cent	
	A.S.T.M. Specimen, Fig. 7	LTB-1 Bar, Fig. 8	A.S.T.M. Specimen, Fig. 7	LTB-1 Bar, Fig. 8
1	36 400	39 500	23.6[a]	39.6
2	32 700	38 300	16.6[a]	32.5
3	36 500	37 900	30.1[a]	33.9
4	36 800	39 400	26.7	35.9
5	34 300	38 900	21.3[a]	39.2
6	38 400	39 500	28.2[a]	31.0
7	32 800	39 700	19.7[a]	34.2
8	38 600	39 700	27.3[a]	35.7
9	38 400	40 100	34.1	37.2
10	37 100	39 300	28.5[a]	33.2
Avg.	36 200	39 230	25.6	35.2

[a] Broke beyond gage marks.

Summary and Conclusions:

A test bar of bronze should provide a means to measure the quality of the metal entering the molds. To this end it should be adequately gated and fed, in molds of the same sand as is used for the castings. Any devices to accelerate the cooling rate of the metal unduly (as chills, or possibly cores) should be prohibited. It is desired that the influence of bad melting practices, lack of pyrometry, etc., not be masked.

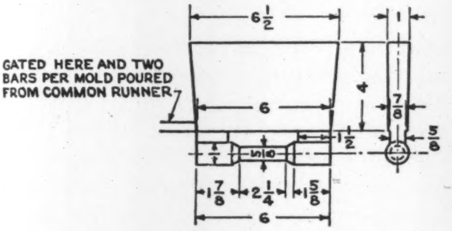

FIG. 17.—Test Bar Prescribed in Fig. 10A of Federal Specification QQ-M-151a.

The bar used also should be economical to cast, machine, and test.

The A.S.T.M. specimen, Fig. 7, is improperly gated for alloys such as covered by A.S.T.M. Specifications B 61 and B 62. It also is uneconomical to cast, machine, and test. These objections apply also to the Federal test bar shown in Fig. 10 (dimensions similar to the A.S.T.M. specimen) and quite probably to Fig. 10A of Federal Specification QQ-M-151a which appears in Fig. 17.

The test bar shown in Fig. 16 (Federal Specification QQ-M-151a, Fig. 11A) is economical but is not too well fed. This results in low tensile strength and ductility and, especially, considerable variation in test results. For purposes of arbitration tests and also for careful control work, long cylindrical ends gripped in V-grips are objectionable.

The Lunkenheimer control bar LTB-1 combines economical production and testing with an adequate gating and riser system, clean well-fed bars being a result. It is well suited for arbitration test purposes, and also reflects variations in foundry practice. This bar is recommended to Subcommittee X, of Committee B-5, for an "arbitration" bar. The realistic demands of war indicate that it would be desirable to offer this bar to the Government authorities for Federal Specification QQ-M-151a.

Acknowledgment:

These data are offered by The Lunkenheimer Co. as a contribution to the war effort of A.S.T.M. Committee B-5. Among those whose help, care, and accuracy have made this paper possible are Messrs. S. A. Weigand, E. L. Bunting, H. L. Montgomery, and A. J. Smith of The Lunkenheimer Co.

COMPARISON OF A.S.T.M. AND O.B. TEST BARS

BY FRED L. WOLF[3]

In Fig. 18 is shown a sketch of the O.B. (Ohio Brass Co.) test bar which was developed to give sound bars as measured by the deep etch test. The ends of the bar outside the gage length are the minimum length which fit the holders of the testing machine, and two bars are produced from 4¾ lb. of metal. The mold is poured uphill at about 7 deg. to the horizontal. Little machining is needed to prepare the specimens.

Investigation made by the Ohio Brass Co. of the tensile properties of cast red brass having the nominal composition, copper 84 per cent; tin, 3.00 per cent; lead, 3.00 per cent; zinc, 10.00 per cent, as revealed by test bars of O.B. design, and A.S.T.M. design shows that the O.B.

design gives results more uniformly representative of the properties of the metal.

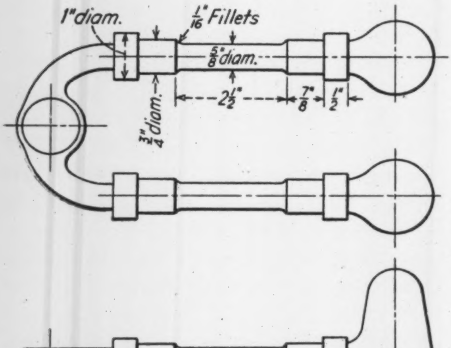

FIG. 18.—O.B. Test Bar Shortened and Re-Gated to Produce Sound Specimens As Shown By Deep-Etch Test.

Tests indicated that the O.B. bar is less affected by conditions of temperature and fluidity of the molten metal than is the A.S.T.M. specimen as reflected by the tensile strength.

	O.B. Bar, Fig. 18	A.S.T.M. Specimen, Fig. 7
Yield point, psi	14 487	14 807
Tensile strength, psi	33 820	32 930
Elongation in 2 in., per cent	29.0	20.5

One interesting effect was noted. The strength of the O.B. test bar does not tend to fall off with the decreasing pouring temperature while that of the A.S.T.M. specimen does. This may be accounted for by the great tendency of the thin gate on the A.S.T.M. coupon to freeze at lower temperatures, thereby decreasing the effectiveness of the riser.

Tensile strengths in order of pouring were as follows:

	Average Values O.B. Bar, Fig. 18	A.S.T.M. Specimen, Fig. 7
Tensile strength, psi	33 435 33 275 34 750	35 800 33 000 33 000

DATA SUBMITTED BY W. E. MARTIN[4]

The data shown in Table VII were obtained on tests of a bronze having the nominal composition 88 per cent copper, 8 per cent tin, and 4 per cent zinc, known also as modified G bronze.

TABLE VII.—RESULTS OF TENSION TESTS ON 88-8-4 BRONZE ALLOY.

Type Bar	Vendor	Number of Bars Tested	Number of Bars Failed	Average Tensile Strength, psi.	Average Yield Strength, psi.	Average Elongation in 2 in., per cent
Machined	A....	94	10	43 000	20 000	36
Machined	B....	114	39	40 000	20 000	23
As cast	A....	57	4	48 000	21 000	39
As cast	B....	22	7	45 000	23 000	25

The machined test bar was substantially the same as Fig. 10A of Federal Specification QQ-M-151a, known as the Crown bar. The bar tested as cast was substantially the same as Fig. 11A of Federal Specification QQ-M-151a, as shown in the accompanying Fig. 16.

The test results in Table VII show in the case of each vendor an advantage for the as-cast test bar of about 5000 psi. tensile strength and 2 to 3 per cent elongation.

The trend of these data confirm data submitted earlier in this report by Messrs. J. J. Curran, J. W. Bolton, and F. L. Wolf.

The bars cast to size showed better properties than those cast in the machined bar. This does not necessarily conflict with the data of Bolton, who shows that the Fig. 10 (Webbert bar) gave better results on a modified alloy than did a cast-to-size bar. The area of riser ingress quite probably has a marked effect. It is just these things that incline us to the opinion that no one bar is suited for a wide range of alloys. The considerable saving in time on the unmachined bar justifies its use.

REPORT OF COMMITTEE B-6
ON
DIE-CAST METALS AND ALLOYS

Committee B-6 on Die-Cast Metals and Alloys held two meetings during the year: one in Chicago, Ill., on June 24, 1941, and one in Philadelphia, Pa., on January 13, 1942, with an attendance of 41 and 21, respectively. Certain of the subcommittees also held other meetings at various times during the year.

A balance of $52.10 is reported by Society headquarters in the fund of Committee B-6 as of December 31, 1941. However, the committee is actually in debt to the extent of $364.20 because a bill for $416.30, incurred in the construction of exposure racks, is still unpaid.

The committee has undertaken to raise $2000 to pay this bill and to have cash available to cover the expenses when the exposure tests come due. The special committee appointed to solicit the contributions consists of G. O. Hiers, chairman, W. M. Peirce, A. E. Keskulla, J. C. Fox, and C. R. Ince.

The following additions and changes in membership have been approved:

M. H. Jameson, Aluminum Co. of America,
E. I. Valyi, Hydraulic Press Mfg. Co.,
A. F. Waltz, Advance Pressure Castings, Inc.,
H. Lindenberger (deceased), U. S. Reduction Co.

At the present time, the committee consists of 66 members, of whom 27 are classified as producers, 20 as consumers, and 19 as general interest members.

EMERGENCY ALTERNATE PROVISIONS

In the interest of conserving aluminum, the committee has recommended Emergency Alternate Provisions in the following specifications:

Tentative Specifications for:

Aluminum-Base Alloy Die Castings (B 85 - 39 T),[1] and
Zinc-Base Alloy Die Castings (B 86 - 41 T).[2]

The Emergency Alternate Provision in Specifications B 85 provides for a new alloy, identified as alloy VIIB, as a substitute for the present alloy No. VII, and the Emergency Alternate Provisions in Specifications B 86 provide a new alloy No. XXIV as a substitute for the present alloy No. XXIII and a new alloy No. XXVI as a substitute for the present alloy No. XXV.

These Emergency Alternate Provisions were accepted by Committee E-10 on Standards on April 6, 1942, and have been published as stickers for attachment to the specifications to which they apply. They have also been published in the ASTM BULLETIN, No. 116, May, 1942.

I. ADOPTION OF TENTATIVE STANDARD AS STANDARD

The committee recommends that the Tentative Specifications for Aluminum-Base Alloy Die Castings (B 85 - 39 T)[1] be revised as follows and approved for reference to letter ballot of the Society for adoption as standard:

Table I.—Replace alloy No. VII by new alloys VIIA and VIIB conforming

to the following requirements as to chemical composition.

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[1] 1939 Book of A.S.T.M. Standards, Part I, p. 1147.

[2] 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 544.

	Alloy VIIA	Alloy VIIB
Copper, per cent	3.5 to 4.5	3.5 to 4.5
Silicon, per cent	4.5 to 7.5	4.5 to 7.5
Nickel, max., per cent	0.3	0.3
Iron, max., per cent	2.0	1.3
Zinc, max., per cent	1.0	0.6
Manganese, max., per cent	0.5	0.5
Magnesium, max., per cent	0.10	0.10
Total other impurities, max., per cent	0.5	0.5
Any one impurity, max., per cent	0.15	0.15
Aluminum	remainder	remainder

This recommendation has been submitted to letter ballot of the committee which consists of 66 voting members; 64 members returned their ballots, of whom 33 have voted affiramtively, 5 negatively, and 26 members marked their ballots "not voting."[a]

II. TENTATIVE STANDARDS CONTINUED AS TENTATIVE

The committee recommends that the following tentative specifications be continued as tentative without revision:

Tentative Specifications for:

Zinc-Base Alloy Die Castings (B 86 – 41 T),[2]
Magnesium-Base Alloy Die Castings (B 94 – 40 T),[3] and
Lead- and Tin-Base Alloy Die Castings (B 102 – 39 T).[4]

In the case of Specifications B 102, action was taken at the June, 1941, meeting to submit this recommendation to letter ballot of the committee since these specifications had been published as tentative for a two-year period. This resulted in the following favorable vote recommending the continuation of these specifications as tentative: From a voting membership of 66, 52 members returned their ballots, of whom 39 voted affirmatively, 0 negatively, and 13 members marked their ballots "not voting."

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Aluminum-Base Die-Casting Alloys (E. E. Thum, chairman) has under consideration, with Subcommittee II on Aluminum Alloy Castings of Committee B-7 on Light Metals and Alloys, the study of the effects of minor alloying elements on aluminum alloys. This task will be pursued by a review of the literature and through questionnaires which are to be sent to the manufacturers of die castings.

The study of an alloy of 8 per cent magnesium, balance aluminum has been delayed first because of certain patent problems and then because of the National Emergency. However, it was pointed out that metal is now available for scientific and technical research and that three manufacturers of die castings have volunteered to prepare the test bars. Therefore, it was decided to proceed with the testing program in the near future.

The Emergency Alternate Provision in the Tentative Specifications for Aluminum-Base Alloy Die Castings (B 85 – 39 T), mentioned earlier in this report, was proposed by this subcommittee in the interest of conserving aluminum during the period of National Emergency. The subcommittee is proposing the addition of new alloys VIIA and VIIB as indicated above in Specifications B 85. New alloy VIIB is that issued as an Emergency Alternate Provision as mentioned earlier in the report with changes in the zinc, tin, and total impurities content. With the adoption of this revision the Emergency Alternate Provisions in Specifications B 85 will be withdrawn.

In view of the accepted embrittling effect of iron in aluminum die castings, Subcommittee I has undertaken the determination of some mechanical properties for cold-chamber cast alloy VIIB in which the iron content will be varied from 0.6 to 2.0 per cent in increments of 0.2 per cent.

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[a] The classified vote on the adoption as standard of the Tentative Specifications for Aluminum-Base Alloy Die Castings (B 85 – 39 T), as revised, was as follows: Affirmative: 16 producers, 10 consumers, 7 general interests; negative: 5 producers, 0 consumers, 0 general interests.

[3] 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 446.

[4] 1939 Book of A.S.T.M. Standards, Part I, p. 1149.

Subcommittee II on Zinc-Base Die-Casting Alloys (C. T. Prendergast, chairman) has recommended the Emergency Alternate Provisions in the Tentative Specifications for Zinc-Base Alloy Die Castings (B 86 – 41 T) providing for two additional alternate alloys designated as alloy No. XXIV and alloy No. XXVI, as mentioned earlier in this report.

Subcommittee III on Tin- and Lead-Base Die-Casting Alloys (G. O. Hiers, chairman) has been inactive because of the scarcity of both lead and tin and due to the lack of interest shown in these alloys as die-casting materials.

Subcommittee IV on Physical Tests of Die Castings (R. L. Templin, chairman) is actively engaged in collecting data on the tests now used by the industry in determining the general satisfaction of die castings.

Subcommittee IV has encouraged the preparation of a paper entitled "Fatigue Testing of Zinc-Base Alloy Die Castings" by E. H. Kelton. This paper is sponsored by the committee and appears as an appendix to this report.[5]

Subcommittee V on Exposure and Corrosion Tests (W. M. Peirce, chairman) has not undertaken any new exposure tests and none of the bars now in process of aging will be returned from the exposure locations for testing prior to 1944.

Subcommittee VI on Finishing Properties of Die Castings (J. C. Fox, chairman) reported that since the organization of Committee B-8 had absorbed the functions of this subcommittee, the only unfinished business consisted of the prepraration of a paper covering finishes for lead- and tin-base alloys. Inasmuch as this paper has not been written and present applications of lead and tin die castings do not require finishes, this project was cancelled. Subcommittee VI was formally discharged with thanks from the committee for the splendid results achieved and for the excellent papers sponsored by this subcommittee.

Subcommittee VII on Magnesium-Base Die-Casting Alloys (J. A. Gann, chairman) still has under consideration a proposed revision of the Tentative Specifications for Magnesium-Base Alloy Die Castings (B 94 – 40 T) to incorporate requirements for a new alloy to be known as alloy No. 13X. However, the subcommittee has not yet released information on this subject to the committee for approval.

The present officers of the committee have been re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 66 voting members; 55 members returned their ballots, of whom 51 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. R. TOWNSEND,
Chairman.

G. L. WERLEY,
Secretary.

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[5] See p. 692.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee B-6 recommended changes in the Emergency Alternate Provision in Tentative Specifications for Zinc-Base Alloy Die Castings (EA – B 86), referred to in this report, which were accepted by Committee E-10 on Standards on October 19, 1942, and on December 31, 1942. The revised emergency provisions were published in ASTM BULLETIN, No. 119, December, 1942, under the designation EA – B 86a, and have been issued in the form of a pink sticker for attachment to the specifications.

Committee B-6 also submitted to Committee E-10 proposed Specifications for Special Grade Zinc-Base Alloy Die Castings, which were accepted by Committee E-10 on October 28, 1942, for publication as tentative and assigned the designation B 186 – 42 T. The new tentative specifications appear in the 1942 Book of A.S.T.M. Standards, Part I, p. 1428.

On November 27, 1942, Committee E-10 accepted the proposed Specifications for Copper Base (Brass) Alloy Die Castings, submitted by Committee B-6, for publication as tentative. The new tentative specifications have been assigned the designation B 176 – 42 T and appear in the 1942 Book of A.S.T.M. Standards, Part I, p. 1431.

REPORT OF COMMITTEE B-7

ON

LIGHT METALS AND ALLOYS, CAST AND WROUGHT

Committee B-7 on Light Metals and Alloys, Cast and Wrought, has held but one meeting during the past year, in Chicago, Ill., on June 23, 1941.

Several changes in membership have been made during the year including the loss of several members and the addition of several others. We record with regret among these changes, the loss, through death, of Henry Lindenberger, U. S. Reduction Co., East Chicago, Ind. The membership of the committee now totals 48, of whom 22 are classified as producers, 17 as consumers, and 9 as general interest members.

As expected under present conditions, committee activity has been slowed down materially as a result of the pressure on its membership due to the War Emergency. A demand for relaxed limits to permit the use of a maximum amount of secondary and lower grade materials has developed. In this connection Committee B-7 is recommending certain changes at this time in several of its specifications. As soon as this situation is more fully defined, additional recommendations as may be required will be proposed as Emergency Alternate Provisions.

Committee B-7 has devised a code system for the classification and designation of the aluminum-base alloys. The basis of this code system is as follows:

1. One or two letters will be used to designate the major alloying elements. If there is but one major alloying element, only one letter will be used. If there are two major alloying elements present, two letters will be used, the first letter designating that element present in the larger amount.
2. Numbers will be used following the letters which further define the number of alloying elements. If this number is below 20, it will indicate that only those elements designated by the code letters are present in the alloy. If this number is between 20 and 40, it will indicate that there is one alloying element present other than those indicated by the code letters. If the number is between 40 and 60, it will indicate that there are two alloying elements present other than those indicated by the code letters. Similarly, a number between 60 and 80 will indicate three additional alloying elements and so on.
3. Only those elements are considered alloying elements that are intentionally added and are therefore defined by both minimum and maximum limits in the specification.
4. The code letters to be used are as follows:

A—aluminum
C—copper
G—magnesium
M—manganese
N—nickel
R—chromium
S—silicon
Z—zinc

The use of the letter "A" for aluminum is necessary since various grades and purities are available in both ingot and wrought forms.

The application of this code system to the various alloys under the jurisdiction of Committee B-7 will be made this year by appropriate editorial changes in the specifications.

Revisions of Tentative Standards

The committee recommends that the following tentative specifications be revised as indicated below and continued as tentative:

Tentative Specifications for Aluminum for Use in Iron and Steel Manufacture (**B 37 – 41 T**):[1]

Table I.—Change from its present form to read as follows:

TABLE I.—CHEMICAL REQUIREMENTS.

Grade	Aluminum, min., per cent by difference	Copper, max., per cent	Zinc, max., per cent	Magnesium, max., per cent	Total of All Impurities,[a] Including Iron, Silicon, Manganese, max., per cent
98	98.0	0.2	0.2	0.5	2.0
94 to 96	94.0	4.0	1.0	1.0	6.0
90 to 94	90.0	6.5	2.0	2.0	10.0
82.5 to 90	82.5	8.0	2.5	2.5	17.5

[a] Commercial grades of aluminum may also contain small amounts of one or more of the following elements: nickel, lead, tin, bismuth, chromium, molybdenum, and titanium. These minor elements need not be determined chemically or spectrographically unless otherwise specified in the contract or order.

Tentative Specifications for Aluminum-Base Alloys in Ingot Form for Sand Castings (**B 58 – 41 T**):[2]

Table I.—Add a new alloy ZG41 conforming to the following requirements as to chemical composition:

	Alloy ZG 41
Copper, max., per cent	0.2
Iron, max., per cent	0.8
Silicon, max., per cent	0.3
Magnesium, per cent	0.5 ± 0.1
Zinc, per cent	$5.0\left\{\begin{matrix}+0.7\\-0.2\end{matrix}\right.$
Titanium, per cent	0.2 ± 0.1
Chromium, per cent	0.5 ± 0.1
Total other impurities, each max., per cent	0.05
Aluminum	remainder

Tentative Specifications for Aluminum-Base Alloys in Ingot Form for Die Castings (**B 125 – 39 T**):[3]

Table I.—Change alloy VII to conform to the following requirements:

	Alloy VII
Copper, per cent	3.5 to 4.5
Silicon, per cent	4.5 to 7.5
Nickel, max., per cent	0.3
Iron, max., per cent	1.0
Zinc, max., per cent	0.5
Manganese, max., per cent	0.5
Magnesium, max., per cent	0.10
Total other impurities, max., per cent	0.5
Single impurity not specifically mentioned, max., per cent	0.15
Aluminum	remainder

Tentative Specifications for Aluminum-Base Alloy Sand Castings (**B 26 – 41 T**):[4]

Table I.—Change the silicon requirement for alloy O (new alloy ZG41) from "0.6 max. per cent" to "0.4 max. per cent."

Production test results indicated that this change is necessary if the mechanical property requirements are to be met.

On the recommendation of Subcommittee III on Wrought Aluminum and Wrought Aluminum Alloys, the committee recommends that the following six specifications be revised as indicated below:

Tentative Specifications for:

Aluminum Sheet and Plate (B 25 – 41 T),[5]
Aluminum-Alloy (Duralumin) Sheet and Plate (Aluminum-Copper-Magnesium-Manganese) (B 78 – 41 T),[6]
Aluminum-Manganese Alloy Sheet and Plate (B 79 – 41 T),[7]
Aluminum-Alloy (Duralumin) Bars, Rods, Wire, and Shapes (Aluminum-Copper-Magnesium-Manganese) (B 89 – 41 T),[8]
Aluminum-Magnesium-Chromium Alloy Sheet and Plate (B 109 – 41 T)[9] and
Aluminum-Manganese Alloy Sheet and Plate for Use in Welded Pressure Vessels (B 126 – 40 T).[10]

Section 3.—Add the following requirement for permissible zinc content in the tabulation of chemical composition requirements:

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[1] 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 308.

[2] *Ibid.*, p. 312.

[3] 1939 Book of A.S.T.M. Standards, Part I, p. 1011.

[4] 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 314.

[5] 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 326.

[6] *Ibid.*, p. 335.

[7] *Ibid.*, p. 339.

[8] *Ibid.*, p. 331.

[9] *Ibid.*, p. 344.

[10] 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 329.

Zinc, max., per cent 0.10

Change the requirement for other elements from its present form to read as follows:

Other elements, max., per cent $\begin{cases}\text{each .... 0.05}\\ \text{total .... 0.15}\end{cases}$

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
REVISION OF TENTATIVE STANDARDS			
Spec. for Aluminum Sheet and Plate (B 25 – 41 T)	20	0	0
Spec. for Aluminum for Use in Iron and Steel Manufacture (B 37 – 41 T)	20	0	0
Spec. for Aluminum-Base Alloys in Ingot Form for Sand Castings (B 58 – 41 T)	20	0	0
Spec. for Aluminum-Base Alloy Sand Castings (B 26 – 41 T)	33	0	8
Spec. for Aluminum-Alloy (Duralumin) Sheet and Plate (Aluminum-Copper-Magnesium-Manganese) (B 78 – 41 T)	20	0	0
Spec. for Aluminum-Manganese Alloy Sheet and Plate (B 79 – 41 T)	20	0	0
Spec. for Aluminum-Alloy (Duralumin) Bars, Rods, Wire, and Shapes (Aluminum-Copper-Magnesium-Manganese) (B 89 – 41 T)	20	0	0
Spec. for Aluminum-Magnesium-Chromium Alloy Sheet and Plate (B 109 – 41 T)	20	0	0
Spec. for Aluminum-Base Alloys in Ingot Form for Die Castings (B 125 – 39 T)	20	0	0
Spec. for Aluminum-Manganese Alloy Sheet and Plate for Use in Welded Pressure Vessels (B 126 – 40 T)	20	0	0

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 48 members, with the results shown in Table I.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Aluminum and Aluminum Alloy Ingots (A. Sugar, chairman).—This subcommittee has confined its activities during the past year to a revision of the Tentative Specifications for Aluminum for Use in Iron and Steel Manufacture (B 37 – 41 T). The addition of certain lower grades of aluminum to these specifications seems advisable as recommended earlier in this report.

Revisions in alloy VII of the Tentative Specifications for Aluminum-Base Alloys in Ingot Form for Die Castings, presented earlier in this report, are in conformity with changes being recommended by Committee B-6 on Die-Cast Metals and Alloys in the A.S.T.M. specifications for aluminum die castings.

The addition of a new alloy ZG41 to the Tentative Specifications for Aluminum-Base Alloys in Ingot Form for Sand Castings (B 58 – 37 T) has been approved.

Subcommittee II on Aluminum Alloy Castings (C. H. Greenall, chairman).—As mentioned earlier in this report a reduction in the allowable silicon content for alloy O appearing in the Tentative Specifications for Aluminum-Base Alloy Sand Castings (B 26 – 41 T) has been recommended by this subcommittee in order that the mechanical properties may be met.

The study of the effect of impurities on the characteristics of aluminum alloy castings is being conducted by a subgroup. A valuable contribution has been made by W. Bonsack and is to be published in an early issue of the ASTM BULLETIN.[11]

Subcommittee III on Wrought Aluminum and Wrought Aluminum Alloys (P. V. Faragher, chairman).—This subcommittee has approved revisions in six specifications under its jurisdiction as presented earlier in this report.

Subcommittee IV on Magnesium and Magnesium Alloys, Cast and Wrought (J. A. Gann, chairman).—This committee has reported no activity during the past year.

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[11] ASTM BULLETIN, No. 117, August, 1942, p. 45.

Subcommittee V on Testing Light Alloys (R. L. Templin, chairman).—A review of the illustrations of the ½ in. diameter tension test specimens appearing in the various B-7 specifications has brought to light some dimensional discrepancies. These have been called to the attention of Committee E-1 on Methods of Testing and a revised sketch has been prepared. Since this change in the illustration of the test specimen involves no revision in the requirements of the various specifications but merely presents those requirements more clearly, it is being handled editorially.

Subcommittee VI on Anodic Oxidation of Aluminum and Aluminum Alloys (J. D. Edwards, chairman).—Progress has been reported in connection with the program designed to compare the performance of anodic coatings in the salt spray test with their behavior under atmospheric exposure. Six laboratories are cooperating in these tests. A progress report of the results should be available for presentation at the next annual meeting.

The subcommittee has reviewed the Tentative Methods of Test for Dielectric Strength of Anodized Aluminum (B 110 – 39 T), Test for Sealing of Anodically Coated Aluminum (B 136 – 40 T), and Test for Weight of Coating on Anodically Coated Aluminum (B 137 – 40 T) and recommends that they be continued as tentative.

Special Subcommittee on Classification and Designation of Aluminum Alloys (I. V. Williams, chairman).—Several minor points in connection with the revised nomenclature system for light alloys have been clarified and the code system has been approved as outlined earlier in this report. The subcommittee will attempt to develop a similar system for the magnesium-base alloys.

The present officers of the committee were re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 48 members; 41 members returned their ballots, of whom 37 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

SAM TOUR,
Chairman.

H. J. ROWE,
Secretary.

REPORT OF COMMITTEE B-8
ON
ELECTRODEPOSITED METALLIC COATINGS

Committee B-8 on Electrodeposited Metallic Coatings held its organization meeting on March 6, 1941, in Washington, D. C. Much of its activity during the current year was concerned with perfecting its organization and studying the needs for plating specifications. Five subcommittees have been appointed and brief reports of their work are given below. The committee held two meetings during the year: one on June 25, 1941, in Chicago, Ill., and one on January 13, 1942, in Philadelphia, Pa. The subcommittees met in Chicago, Ill., on October 1, 1941, and in Philadelphia, Pa., on January 13, 1942.

By-laws were adopted upon approval by letter ballot vote as follows: From a membership of 29, 28 members voted affirmatively and one member marked his ballot "not voting."

The committee has approved the scope of activities and functions of the new Advisory Committee on Corrosion by letter ballot vote as follows: From a membership of 37, 36 members voted affirmatively and 1 negatively.

Committee B-8 has not seen fit to recommend any emergency provisions in the existing specifications under its jurisdiction due to the fact that non-defense plating has substantially ceased and there appears to be no good reason for reducing requirements for such coatings as continue to be used on essential defense work.

The great reduction in commercial use of electrodeposited coatings combined with heavy new demands on its individual members in connection with the defense program will result in Committee B-8 being less productive but not idle in its field for the duration of the war. Committee B-8 has expressed to the Executive Committee of the Society and to the Government its readiness and desire to cooperate in the war effort by obtaining and furnishing information relative to electrodeposited coatings and is maintaining its organization for this purpose.

Upon request, Committee B-8 is now represented on the following committees:

Committee D-1 on Paint, Varnish, Lacquer, and Related Products, by B. H. McGar,
Committee D-12 on Soaps and Other Detergents, by G. B. Hogaboom,
Committee D-19 on Water for Industrial Uses, by C. Kocour,
Committee E-1 on Methods of Testing, by C. E. Heussner,
Technical Committee VIII on Thickness Measurement, by A. K. Graham, and
Technical Committee XIII on Hydrogen Ion Determinations, by L. R. Westbrook.

The Tentative Methods of Test for Local Thickness of Electrodeposited Coatings (A 219 - 40 T), under the joint jurisdiction of Committee B-8 and Committee A-5 on Corrosion of Iron and Steel are being continued as tentative without revision.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Specifications and Definitions (Gustaf Soderberg, chairman).—A number of subgroups have been organized as follows: Definitions (Wm. Blum, chairman), Lead and Lead

Alloy Coatings (Thor H. Westby, chairman), Tin Coatings (H. A. Pray, chairman), Silver Coatings (A. K. Graham, chairman), Heavy Chromium Coatings (Glenn R. Carley, chairman).

Of these subgroups, those on Definitions and on Heavy Chromium Coatings[1] have reported definite progress.

Subcommittee II on Performance Tests (C. E. Heussner, chairman).—One subgroup on Collection and Correlation of Data on Outdoor Exposure Tests (Wm. Blum, chairman) has been appointed.

Subcommittee III on Conformance Tests (C. H. Sample, chairman).—Subgroups on Thickness Tests (W. L. Pinner, chairman), Porosity Tests (J. E. Fritts, chairman) and Tests of Structural Properties (C. H. Sample, chairman *pro tem*) have been organized.

Subcommittee IV on Electroplating Practice (A. K. Graham, chairman).—Subgroups on Preparation of Base Metals, Plating Procedures for Specific Base Metals, and Procedures for Stripping and Replating have been authorized. The first-mentioned subgroup is collecting data on the preparation of cold-rolled steel for electroplating.

Subcommittee V on Supplementary Protective Finishes for Metallic Coatings (B. H. McGar, chairman).—This subcommittee has collected data on the use of oil, wax, and lacquer coatings, and passive films and is engaged in the study of these data.

The present officers of the committee have been re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which now consists of 51 members; 47 members returned their ballots, of whom 44 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

E. M. BAKER,
Chairman.

GUSTAF SODERBERG,
Secretary.

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[1] See Editorial Note, below.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee B-8 presented to the Society through Committee E-10 on Standards a new Tentative Recommended Practice for Chromium Plating on Steel for Engineering Use. This recommended practice was accepted by Committee E-10 for publication as tentative on March 6, 1943, and is available in separate reprint form under the designation B 177 - 43 T.

REPORT OF COMMITTEE C-1

ON

CEMENT

Committee C-1 on Cement held two meetings during the year, both meetings being very well attended by members and also by interested visitors. One of the meetings was so located that an opportunity was afforded for those in attendance to visit numerous nearby laboratories and cement mills. The other meeting was specially called to consider cement questions arising out of the present war emergency—questions relating to temporary modifications of the requirements in the A.S.T.M. Standard Specifications for Portland Cement (C 150 - 41), simplification of arrangements for testing and inspection of cement, number of types of cement, etc.

At the last named meeting there were present representatives of the War Production Board, including J. L. Haynes, Chief of the Lumber and Building Materials Branch. The committee, therefore, had the benefit of Mr. Haynes' explanations concerning some of the numerous items which were discussed. As a result of that meeting, Committee C-1 offered to the War Production Board a number of recommendations concerning specifications for and inspection of portland cement. Also, Committee C-1 appointed a special subcommittee, under the chairmanship of J. L. Miner, authorized to offer, on its own initiative, recommendations to the War Production Board regarding reduction in number of types of cement, emergency provisions in A.S.T.M. Specifications C 150, limitations on types of cement which should be made in any certain locality, etc. This subcommittee was also to act, as provided under the A.S.T.M. Regulations Governing Standing Committees, to prepare, if thought needed, Emergency Alternate Provisions and Emergency Specifications for submission to Committee E-10 on Standards for issue by the Society for use during the emergency. The personnel of this subcommittee was selected to afford appropriate distribution of interest and also of geographical location. This special subcommittee prepared Emergency Alternate Specifications for Portland Cement which were presented to the Society and accepted by Committee E-10 on Standards on June 5, 1942, and issued under the designation EA - C 150.[1]

SUBCOMMITTEE ACTIVITIES

Throughout the year a very considerable volume of subcommittee work was maintained. Some of this current work has not reached the stage where lengthy or conclusive reports were possible, but progress is being made.

Chemical Analysis:

The Working Committee on Methods of Chemical Analysis, under the chairmanship of W. C. Hanna, has continued its customary year-round activity. Based on extensive cooperative study during the year, the subcommittee presented[2] a recommended tentative alternate method for the rapid determination of sodium oxide and potassium oxide in cement.[3] The subcommittee also presented a recommended tentative method for determining sulfide sulfur,[3] as the result of work performed during the preceding year. Committee C-1 has approved the two foregoing recommendations of the Working Committee and is now presenting those recommendations to the Society as proposed revisions in the Tentative Methods of Chemical Analysis of Portland Cement (C 114 - 40 T).

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[1] These Emergency Alternate Specifications appear in the 1942 Book of A.S.T.M. Standards, Part II, p. 1017.

This Working Committee has also recently begun an additional study of the method published by R. B. Rudy for "Determination of Sulfuric Anhydride in Portland Cement by Means of the Wagner Turbidimeter."[4] This method was studied briefly by the Working Committee some years ago, and has also been used by some laboratories. Recent developments showed the desirability of studying the problem of preparing a detailed description of the method in proper form for presentation as a proposed A.S.T.M. quick alternate method. Rapid progress has been reported in the current study of certain of the details and the endeavor to learn more about the nature of the reproducibility of results among different laboratories. Guided by the results of that work, Committee C-1 is recommending, for acceptance as tentative, an alternative rapid method for determining sulfur trioxide in cement by the turbidimeter.[3]

This Working Committee has also prepared a proposed revision of the requirements for balances and weights now prescribed in Section 3 of the Standard Methods of Chemical Analysis of Portland Cement (C 114 - 40) to bring them into conformity with the requirements for analytical balances as contained in Federal Specification SS-C-158a, and also to present additional information about weights. Committee C-1 now presents the following revision in Section 3 for immediate adoption and accordingly asks for a nine-tenths affirmative vote in order that this change in Standard Methods C 114 - 40 may be submitted to letter ballot of the Society.

Section 3.—Change from its present form to read as follows:

3. (*a*) *Balances.*[a]—The analytical balances used in the chemical determinations shall conform to the following requirements:

Capacity not less than 100 g. in each pan,

The two arms of the beam equal within (*1*) two parts in 100,000 for balances in use or (*2*) one part in 100,000 for new balances (Note 1),

Capable of reproducing results within 0.0002 g., and

Sensibility reciprocal[b] not more than 0.0005 g. per division of the graduated scale for any load up to 100 g. (Note 2). In case of direct-reading balances this requirement does not apply.

Any rapid weighing device that may be provided, such as a chain, damped motion, or heavy riders, shall not introduce errors greater than 0.0001 g. at any reading and with any allowable load on the scale pans.

NOTE 1.—The lengths and ratios of the arms may change with use, and with age if not used.

NOTE 2.—If an auxiliary metal or glass pan is used, its weight is a part of the load.

(*b*) *Weights.*[a]—The weights used in the chemical determinations shall conform to the requirements of the National Bureau of Standards specifications for class "S" weights as prescribed in Bureau of Standards *Circular No. 3*, "Design and Test of Standards of Mass." The weights (including the riders) shall be checked when questioned, or at least once a year, with weights that have been certified by the National Bureau of Standards within five years. They shall be adjusted within the tolerances given in the above mentioned specifications for class "S" weights, according to their apparent mass as determined by comparison with brass standards in air.

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[2] W. C. Hanna, L. N. Bryant, and T. A. Hicks, "Investigation of Quick Methods for Determining Sodium Oxide and Potassium Oxide in Portland Cement," ASTM BULLETIN, No. 114, January, 1942, p. 41.

[3] These methods were accepted by the Society and have been incorporated in the Tentative Methods of Chemical Analysis of Portland Cement (C 114 - 42 T), which appear in their latest revised form in the 1942 Book of A.S.T.M. Standards, Part II, p. 1058.

[4] *Journal of Research*, Nat. Bureau Standards, Vol. 16, No. 6, p. 555, (1936) (*Research Paper 893*).

The Working Committee also offered, as a result of a recently completed cooperative study by a number of laboratories, a revision of the standard method for determining insoluble residue in cement. Committee C-1 recommends this revision for immediate adoption and accordingly asks for a nine-tenths affirmative vote in order that this modification in Section 31 of Standard Methods C 114 - 40 may be referred to letter ballot of the Society.

Section 31.—Change from its present form to read as follows:

31. To 1 g. of the sample, add 10 ml. of cold water and 5 ml. of HCl (sp. gr. 1.18). Warm the solution gently and grind the material with the flattened end of a glass rod for a few minutes until it is evident that decomposition of the cement is complete (Note). Dilute the solution to 50 ml. and digest for 15 min. at a temperature just short of boiling. Filter the residue, wash six times with hot water, and digest the filter paper and contents in 100 ml. of NaOH solution (1 per cent), the solution being held at a temperature just short of boiling for 15 min. Using methyl red as indicator, acidify the solution with HCl and add an excess equivalent to 4 or 5 drops of HCl (sp. gr. 1.18). Filter and wash the residue 12 to 15 times with hot NH_4Cl solution (2 per cent). Ignite the residue in a tared crucible at 900 to 1000 C., cool it in a desiccator, and report the gain in weight as insoluble residue.

NOTE.—If a sample of portland cement contains an appreciable amount of manganic oxide, there may be brown compounds of manganese which dissolve slowly in cold diluted HCl but rapidly in hot HCl in the specified strength. In all cases, dilute the solution as soon as decomposition is complete.

Interested laboratories have contributed their efforts in studying the possibilities of applying spectrographic methods in the quantitative analysis of cements. These studies are being continued.

Alkali (Na_2O, K_2O) in Cement:

In its report for 1941, Committee C-1 explained in some detail the reasons for attaching great importance to the current study of the question of specification limits for Na_2O and K_2O in portland cements. While no recommendations are now being made regarding this matter, this special subcommittee which had been appointed to study this subject has been continuously active under the chairmanship of R. F. Blanks.

Volume Change and Soundness:

The Working Committee on Volume Change and Soundness, under the chairmanship of H. F. Gonnerman, has actively continued the study of questions under its jurisdiction. However, no changes in the Method of Test for Autoclave Expansion of Portland Cement (C 151 - 40 T) are now proposed, and it is recommended that this method be retained as tentative without revision. Arrangements were made for the Cement Reference Laboratory to collect certain desired data on apparatus.

Fineness:

The Working Committee on Fineness (P. H. Bates, chairman) has continued its cooperative studies of the possibilities of the air permeability measurement method for the determination of the fineness of cement. The results have been encouraging. This subcommittee also recommended that the Tentative

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[a] Information on Analytical Balances and Weights, to supplement the requirements for balances and weights prescribed in these methods, has been prepared and is published as information, not a part of these methods, see the compilation of "A.S.T.M. Standards on Cement," issued as a separate publication, October, 1942, p. 40.

[b] The sensibility reciprocal is a measure of the sensitiveness of a balance and is the weight required to move the position of equilibrium of the pointer one division. For a complete definition of sensibility reciprocal, see National Bureau of Standards *Handbook H22*, "Specifications, Tolerances, and Regulations for Commercial Weights and Measures and Weighing and Measuring Devices," pp. 68 and 69.

Method of Test for Fineness of Portland Cement by Means of the Turbidimeter (C 115 - 41 T) be adopted as standard. Committee C-1 has approved that proposal and is now recommending its adoption by the Society.

Strength:

The Working Committee on Strength (O. L. Moore, chairman) has continued its study of problems relating to its work. The question of increasing the allowable size of batches, for both tensile strength and compressive strength tests, is receiving attention. The subcommittee is also considering details of the Tentative Method of Test for Compressive Strength of Portland-Cement Mortars (C 109 - 37 T), devoting particular attention to requirements for apparatus. It is expected that these studies will result in early proposals concerning revisions in details of that method. However, pending the completion of those studies, Committee C-1 recommends that Tentative Method C 109 - 37 T be retained without change for another year.

Additions to Cement:

The 1941 Report of Committee C-1 presented reasons for devoting attention to such additions to cement as seem to incorporate a small percentage of air in the concrete. There has been continued interest in the excellent resistance to frost action of concrete made with cement containing certain of such additions. The Working Committee on Additions (H. J. Gilkey, chairman) completed the study, which had been undertaken in the preceding year, on one of these additions when used in portland cements employed in highway paving concrete subject to severe frost action. The Sponsoring Committee on Portland Cements (C. E. Wuerpel, chairman) was instructed to prepare and submit to Committee C-1 appropriate specifications for "treated" portland cements for highways, thus caring for the addition which had just been studied, and providing a suitable specification basis for such other additions of like type as might later be declared acceptable to the committee. The Working Committee on Additions is continuing its studies of additions, particularly including some of the nonproprietary nature.

Blended Cements:

The Sponsoring Committee on Blended Cements (R. E. Roscoe, chairman) has continued the active consideration of its problems, including the question of specifications for portland-blast furnace slag cement. Proposed tentative specifications for portland-pozzuolana cement have been distributed to Committee C-1 for comments. The subcommittee is giving attention to the study of tests for pozzuolanic activity and long-time tests.

Sulfate Resistance:

The Working Committee on Sulfate Resistance (D. G. Miller, chairman) is continuing its studies. Six laboratories have been participating in a program of short-time tests on the 132 cements on which long-time sulfate resistance tests had been made. Additional laboratories may assist in the work. It is too early for detail progress reports on this work.

Portland Cement Specifications:

The Sponsoring Committee on Portland Cements (C. E. Wuerpel, chairman) has been studying certain details of the Standard Specifications for Portland Cement (C 150 - 41)[5] contemplating revisions therein in order to effect desired clarification and more effectively describe the intent of the specifications.

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[5] 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 1.

As a result of those studies, the Sponsoring Committee has prepared the following two revisions for immediate adoption in Specifications C 150 - 41, and Committee C-1 accordingly asks for the necessary nine-tenths affirmative vote in order that this recommendation may be referred to letter ballot of the Society:

Section 3.—Change the definition of portland cement from its present form to read as follows:

3. *Portland Cement.*—Portland cement is the product obtained by pulverizing clinker consisting essentially of hydraulic calcium silicates, to which no additions have been made subsequent to calcination other than water and/or untreated calcium sulfate, except that additions not to exceed 1.0 per cent of other materials may be interground with the clinker at the option of the manufacturer, provided such materials in the amounts indicated have been shown to be not harmful by tests carried out or reviewed by Committee C-1 on Cement.[a]

NOTE.—Tests to determine whether a proposed addition is not harmful will be carried out or reviewed by Committee C-1 on Cement, for those making requests. In the year following that in which an addition has been declared not harmful by the committee, the name of the addition and the amount permitted shall appear as an addendum to Section 3 in a revision of the specifications.

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[a] The committee has declared as not harmful the inclusion of the material known commercially as TDA (composed of triethanolamine and highly purified soluble calcium salts of modified lignin sulfonic acids), manufactured by the Dewey and Almy Chemical Co., when added in an amount not exceeding 0.043 per cent by weight of the cement, except that in type III cement a maximum of 0.08 per cent by weight may be used.

Table II.—Change the third sentence of footnote *c* from its present form: namely,

Tests at 28 days on types I and II cement may be waived at the option of the purchaser.

to read as follows:

Unless otherwise specified, the compressive and tensile strength tests on types I and II cement will be made only at 3 and 7 days.

These changes in Table II will bring the A.S.T.M. specifications into accord with the existing Federal Specification.

This subcommittee has also studied the problem of specifications for certain additions in portland cement, as related earlier in this report under the heading "Additions to Cement." Guided by these studies, the Sponsoring Committee has recently submitted proposed Tentative Specifications for Treated Portland Cement for Concrete Pavements.[6] Committee C-1 recommends that these specifications be accepted for publication as tentative.

Numerous questions within its scope of activity were also considered by this subcommittee.

Miscellaneous Subcommittee Work:

Progress was reported in the activities of certain working committees which have not, however, reached the point where very definite reports were available. Among these were the Working Committee on Bleeding, Plasticity and Workability (F. O. Anderegg, chairman), Working Committee on Heat of Hydration (G. J. Durant, chairman), Working Committee on New Tests (N. T. Stadtfeld, chairman), and Working Committee on Time of Setting (E. E. Berger, chairman).

Cement Reference Laboratory:

Under the supervision of the Subcommittee on Cement Reference Laboratory (G. E. Warren, chairman), the Cement Reference Laboratory at the National Bureau of Standards continued its activities throughout the year. The work of this joint project of the Bureau and the A.S.T.M., under the sponsorship of Committee C-1, is summarized as follows for the period July 1, 1941, to June 30, 1942.

Work was continued on the seventh inspection tour among cement testing laboratories, 163 laboratories being visited. This tour had been started at the end of 1940. Requests for inspection were received from 281 laboratories. The number of inspection requests in the sixth tour was 290.

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[6] These specifications were accepted as tentative by the Society and appear in the 1942 Book of A.S.T.M. Standards, Part II, p. 1055.

The Public Roads Administration continued to require Reference Laboratory inspections at laboratories which test cement for certain Government projects. Copies of reports of numerous inspections have been furnished to that office under that arrangement. In addition to requiring inspections as above, the Public Roads Administration gave financial support to the Reference Laboratory, thus enabling the travel to be continued at the desired rate through the recent months.

The verification of compression testing machines and the examination of the 2-in. cube molds was begun with the current tour. Verification tests were made on 92 compression testing machines, while information was gathered on an additional 53 machines which were not verified because of such reasons as too large capacity for the work involved, insufficient clearance for insertion of proving equipment, lack of suitable upper bearing block for proving ring, absence of operator. Thus, the Reference Laboratory now has data resulting from its verification of more than 120 compression testing machines, with miscellaneous information concerning some of the details of about 60 machines which it did not test. This information is being furnished to the Subcommittee on Strength. To that same subcommittee was sent the information collected on the practice of many laboratories throughout the country with regard to the size of batch used in the preparation of the tension specimens, also the data obtained in the tests of cube molds.

In the absence of a suitable complete book on the subject, the laboratory prepared, for the use of its inspectors, a revision of its booklet of instructions covering the use of proving rings in the verifications of compression testing machines. This verification work has proved to be of continued interest since its start. Indirectly, the work has in some cases proven of value in fields other than cement testing, it being understood that some of the verified machines were used also for testing miscellaneous materials involved in the war effort.

Portions of the comparative test samples Nos. 3, 4, and 5 were sent to a number of laboratories interested in the usual chemical analysis studies and in spectrographic work.

The Working Committee on Methods of Chemical Analysis was furnished with a summary of the data collected on the ignition equipment used by the numerous laboratories which participated in the tests of the Reference Laboratory's comparative samples Nos. 4 and 5.

In the current tour the work has been extended to include demonstrations of autoclave test procedure. In cooperation with the Working Committee on Volume Change and Soundness, data have been assembled regarding details of procedure and performance of apparatus used in the autoclave test. Such data, obtained through inspectors' observations and in reply to a recent questionnaire of the Reference Laboratory, are being summarized for submission to the Working Committee on Volume Change and Soundness.

The field tests of the microammeters used with the Wagner turbidimeter have demonstrated the necessity of frequent check on the performance of these microammeters.

Miscellaneous work has included the training of new personnel, tests of equipment received at the Bureau, caring for inquiries concerning testing equipment and procedure.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARD			
Spec. for Treated Portland Cement for Concrete Pavements	50	4	7
II. REVISION OF TENTATIVE STANDARD			
Chemical Analysis of Portland Cement (C 114 – 40 T):			
New alternate method for the rapid determination of sodium oxide and potassium oxide in cement	55	1	9
New method for determining sulfide sulfur in cement	53	0	10
New alternate rapid method for determining sulfur trioxide in cement by turbidimeter	53	0	6
III. REVISIONS OF STANDARDS, IMMEDIATE ADOPTION			
Spec. for Portland Cement (C 150 – 41)	52	0	7
Chemical Analysis of Portland Cement (C 114 – 40):			
Revision in requirements for chemical balances and weights in Section 3	53	0	6
Revision of procedure in Section 31 for determining insoluble residue	53	0	6
IV. ADOPTION OF TENTATIVE STANDARD AS STANDARD			
Test for Fineness of Portland Cement by Means of the Turbidimeter (C 115 – 41 T)	64	2	6

Recommendations Affecting Standards:

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 82 voting members, with the results shown in Table I.

The following officers were unanimously elected for the ensuing term of two years:

Chairman, P. H. Bates.
Vice-Chairman, C. F. Conn.
Vice-Chairman, D. Wolochow.
Secretary, G. E. Warren.

This report has been submitted to letter ballot of the committee, which consists of 82 voting members; 63 members returned their ballots, of whom 61 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

P. H. BATES,
Chairman.

L. W. WALTER,
Secretary.

REPORT OF COMMITTEE C-7
ON
LIME

Committee C-7 on Lime has held two meetings during the year: on June 25, 1941, in Chicago, Ill., and on May 26, 1942, at Hot Springs, Va. A special meeting of Subcommittee II on Structural Lime was held in Washington, D. C., on December 5, 1941.

Five new members have been added to the committee during the year. At present the total membership is 54, classified as 26 producers, 7 consumers, and 21 general interest members. The year also marked the death of W. E. Carson, vice-chairman of the committee. As president of the Riverton Lime and Stone Co., Riverton, Va., Mr. Carson for many years took an active interest in the affairs of the committee and of the Society.

Subsequent to the 1941 annual meeting, Committee C-7 presented to the Society through Committee E-10 on Standards a tentative revision of the Standard Definitions of Terms Relating to Lime (C 51 - 39). This tentative revision in the form of new Tentative Definitions of Terms Relating to Lime (C 51 - 41 T), was accepted[1] by Committee E-10 on October 23, 1941. Committee C-7 recommends that the new definitions be continued as tentative during the coming year.

I. ADOPTION OF TENTATIVE STANDARD AS STANDARD

Having stood for two years without revision, and in the absence of suggestions for modification, the committee recommends that the Tentative Specifications for Hydraulic Hydrated Lime for Structural Purposes (C 141 - 38 T)[2] be approved for reference to letter ballot of the Society for adoption as standard.

This recommendation has been submitted to letter ballot of the committee which consists of 54 members; 43 members returned their ballots, of whom 33 have voted affirmatively, 0 negatively, and 10 members marked their ballots "not voting."

II. ADOPTION AS STANDARD OF TENTATIVE REVISION OF STANDARD

No comments have been received during the past two years relative to the tentative revision[3] of the Standard Specifications for Quicklime and Hydrated Lime for Silica Brick Manufacture (C 49 - 24), which was presented in the 1940 annual report. The committee therefore recommends that this tentative revision be approved for reference to letter ballot of the Society for adoption as standard.

This recommendation has been submitted to letter ballot of the committee which consists of 54 members; 43 members returned their ballots, of whom 34 have voted affirmatively, 0 negatively, and 9 members marked their ballots "not voting."

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[1] In submitting this recommendation to Committee E-10 on Standards, Committee C-7 reported results of the letter ballot vote as follows: Of a total membership of 49, 32 members returned their ballots, of whom 27 voted affirmatively, 1 negatively, and 4 members marked their ballots "not voting."

[2] 1939 Book of A.S.T.M. Standards, Part II, p. 905.

[3] 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 417.

III. STANDARDS AND TENTATIVE STANDARDS CONTINUED WITHOUT REVISION

The Tentative Methods of Physical Testing of Quicklime and Hydrated Lime (C 110 – 38 T) have stood for two years without revision, and the following standard specifications and methods have not been revised during the past six years:

Standard Specifications for:

Quicklime for Structural Purposes (C 5 – 26),
Hydrated Lime for Structural Purposes (C 6 – 31),
Quicklime and Hydrated Lime for Cooking of Rags in Paper Manufacture (C 45 – 25),
Quicklime for Sulfite Paper Manufacture (C 46 – 27),
Quicklime and Hydrated Lime for Use in the Textile Industry (C 48 – 24) and
Hydrated Lime for Varnish Manufacture (C 47 – 27).

Standard Methods of:

Chemical Analysis of Limestone, Quicklime, and Hydrated Lime (C 25 – 29), and
Sampling, Inspection, Packing, and Marking of Quicklime and Lime Products (C 50 – 27).

The subcommittees have done considerable work on a number of these standards, particularly those covering structural lime and physical and chemical methods of analysis, but none of the investigations have reached a point where definite revisions can be advocated. The other specifications still are considered to be in accord with present practice and require no revision. Committee C-7 therefore recommends to the Society that these standards be continued in their present status.

Subcommittee II on Structural Lime (J. A. Murray, chairman).—The principal subjects under consideration by this subcommittee are two proposed specifications designed to cover the newer types of hydrated lime for structural purposes. One of these is for hydrated lime for mortar purposes and the other for finishing hydrated lime. These hydrates are characterized by the properties of high plasticity and high sand-carrying capacity, and, in the case of the dolomitic types, by substantially complete hydration of the magnesium oxide and low expansion when subjected to steam under pressure.

In this connection, Committee C-7 is attempting to develop a suitable steam pressure method of test to be applied to these newer types of hydrates.

A beginning has been made by the committee toward the drafting of one single master specification for lime used in industrial and chemical processing.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, W. C. Voss.
Vice-Chairman, J. A. Murray.
Vice-Chairman, J. F. Carle.
Secretary, L. K. Herndon.

This report has been submitted to letter ballot of the committee which consists of 54 members; 43 members returned their ballots, of whom 41 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

NATHAN C. ROCKWOOD,
Chairman.

W. VERNON BRUMBAUGH,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee C-7 presented to the Society through Committee E-10 on Standards a tentative revision of the Standard Methods of Chemical Analysis of Limestone, Quicklime, and Hydrated Lime (C 25 – 29). This tentative revision was accepted by Committee E-10 on August 24, 1942, and appears in the 1942 Book of A.S.T.M. Standards, Part II, p. 1439.

REPORT OF COMMITTEE C-8
ON
REFRACTORIES

Committee C-8 on Refractories held two meetings during the year: one on September 4, 1941, in Granville, Ohio, and the other on March 5, 1942, in Cleveland, Ohio. As has been the custom in the past few years, the fall meeting was held in conjunction with the meeting of the Refractories Division of the American Ceramic Society, and the March meeting in conjunction with the spring group meetings of the A.S.T.M.

The committee was very active during the year, and is presenting for the first time specifications for fireclay plastic refractories to be used in the construction of boiler furnaces and incinerators.

At the spring meeting of the committee, consideration was given to the procedure set up by the Society whereby Emergency Alternate Provisions may be promptly issued in order to expedite production or to conserve materials during the period of national emergency. It was the consensus that at the present time there is little need for Committee C-8 to make use of such alternate provisions because, so far at least, the refractories industry is meeting the requirements of industry.

In order to keep all the subcommittees active, the Advisory Committee at the March 5, 1942, meeting discharged the membership of all subcommittees and appointed a chairman for each. These chairmen, with the approval of the Advisory Committee, are to select the members of their committees. Because there is no immediate work for them, Section A on Load, of Subcommittee III, and Section A on Retest Data, of Subcommittee V, have been discharged.

A revision was made in Article VII, Section 1, of the Regulations Governing Committee C-8, permitting discontinuation of standing subcommittees upon letter ballot approval of the committee, on the recommendation of the Advisory Committee.

Two new members were added to the committee during the year: E. E. Callinan, Timken Roller Bearing Co., and Hewitt Wilson, U. S. Bureau of Mines.

The present officers of the committee were re-elected for the ensuing term of two years.

I. NEW TENTATIVE STANDARD

There has been a repeated demand for specifications covering fireclay plastic refractories for boiler furnaces and incinerator service. Committee C-8 has been working on this problem for several years and has developed a set of specifications, and also test methods for determining those properties not covered by other A.S.T.M. standard test methods. The committee recommends that these proposed Specifications for Fireclay Plastic Refractories for Boiler Furnaces and Incinerator Service[1] be accepted for publication as tentative.

II. REVISION OF STANDARD, IMMEDIATE ADOPTION

Methods of Testing Insulating Fire Brick (Compressive Strength, Flexural Strength, and Permanent Linear Change

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[1] These specifications were accepted as tentative by the Society and appear in the 1942 Book of A.S.T.M. Standards, Part II, p. 1141.

After Heating) (**C 93 – 41**).[2]—There is an inconsistency in Section 14 of Methods C 93 – 41. Paragraph (*a*) of this section specifies the calculation of linear shrinkage from measurements taken along the three major axes of the specimen, whereas Paragraph (*c*) specifies the calculation of permanent linear shrinkage after heating from the volume shrinkage as calculated in Paragraph (*b*). The latter is obtained from Paragraph (*a*) and so the linear shrinkage calculated from it (Paragraph (*c*)) would be the same as the weighted average linear change given in Paragraph (*a*). The committee therefore recommends the omission of Section 14 (*c*) from these specifications and asks for a nine-tenths affirmative vote in order that this recommendation may be referred to letter ballot of the Society.

III. ADOPTION OF TENTATIVE STANDARD AS STANDARD

Tentative Definitions of Terms Relating to Refractories (**C 71 – 40 T**).[3]—The committee has cooperated with the Society in drawing up definitions that cover adequately the terms as applied to the refractories industry but still conform to A.S.T.M. requirements. Last year it recommended for adoption as standard the tentative definitions for abrasion, corrosion, erosion, slagging, burning, and calcining as they appear in C 71 – 40 T. Committee E-8 on Nomenclature and Definitions requested that the formal adoption of these definitions as standard be deferred for a year in order that certain editorial suggestions might be reviewed and discussed with the committee. It appeared that if the words "of refractories," (except in the case of calcining, which would read "of refractory materials") were added to the titles of the definitions concerned they would be acceptable to Committee E-8. These editorial changes were approved by Committee C-8. It was also recommended that the definition for erosion be editorially revised by omission of the phrase "such as molten metals and slags."

Committee C-8 accordingly recommends that with these editorial changes the definitions of the terms abrasion, burning, corrosion, erosion, and slagging be approved for reference to letter ballot of the Society for adoption as standard. The definitions in their revised form are given below. It is further recommended that the definition for calcining be retained as tentative.

Abrasion of Refractories.—Wearing away of refractory surfaces by the scouring action of moving solids.

Burning (Firing) of Refractories.—The final heat treatment to which refractory brick and shapes are subjected in the process of manufacture for the purpose of developing bond and other necessary physical and chemical properties.

Corrosion of Refractories.—Destruction of refractory surfaces by the chemical action of external agencies.

Erosion of Refractories.—Wearing away of refractory surfaces by the washing action of moving liquids.

Slagging of Refractories.—Destructive chemical reaction between refractories and external agencies at high temperatures, resulting in the formation of a liquid.

IV. ADOPTION AS STANDARD OF TENTATIVE REVISIONS OF STANDARDS

Method of Test for Pyrometric Cone Equivalent of Refractory Materials (**C 24 – 35**).—The tentative revision[4] of this method, published in 1940, involves: (*a*) a restatement of the scope of the method, (*b*) handling of silica cements, and (*c*) a slight modification in the dimensions of the pyrometric test cones. The committee recommends that this tentative revision be approved for reference to letter ballot of the Society for adoption as standard.

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[2] 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 57.

[3] 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 222.

[4] 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 419.

The committee also calls attention to an editorial correction. In the proposed revision of Section 3 (*a*) of Method C24 the over-all height of the cone is given as 1_{18} in. but should read $1\frac{1}{8}$ in.

Method of Panel Test for Resistance to Thermal and Structural Spalling of Refractory Brick (C 38 – 36).—The tentative revision[5] of this method, published in 1940, involves a new style of back-up insulation for panels during the reheat portion of the spalling test. The object was to devise a means of insulating the panels to effect a uniform heat distribution at the interface of the panel and the insulation. As a result an even spalling takes place over the entire surface of the panel rather than a more or less concentrated action along the center vertical section. The tentative revision also includes a revised drawing of the new design for the insulating back-up brick of the test panel. As no objections have been received since the proposed changes were published, the committee recommends that the tentative revision of Method C 38 – 36 be approved for reference to letter ballot of the Society for adoption as standard.

Method of Panel Test for Resistance to Thermal and Structural Spalling of High Heat Duty Fireclay Brick (C 107 – 40).—The tentative revision[6] of this method, published in 1940, specifies the kind of insulating fire brick to be used in the panels. The committee recommends that this tentative revision of Method C 107 – 40 be approved for reference to letter ballot of the Society for adoption as standard.

Method of Panel Test for Resistance to Thermal and Structural Spalling of Super Duty Fireclay Brick (C 122 – 40).—The tentative revision[6] of this method, published in 1940, is the same as that recommended in Method C 107, namely, a description of the kind of insulating fire brick to be used in the panels. The committee recommends that the tentative revision of Method C 122 – 40 be approved for reference to letter ballot of the Society for adoption as standard

V. STANDARDS CONTINUED WITHOUT REVISION

Standard Method of Test for Particle Size of Ground Refractory Materials (C 92 – 36).—Modifications of this method have been under consideration, and suggestions which have been made are being incorporated. Many of the changes are editorial in nature but several have to do with changes in substance, and, consequently, it will be necessary to issue the new draft as a tentative method. The revision will point out the usefulness of the dry method of sieve analysis. Reconsideration is being given to the size of samples to be used and to features of technique. A separate section will deal with the determination of moisture in the samples for sieve analysis and samples of cold-set mortar, plastic refractories, wet mixes, etc. The committee expects to present the new draft for approval at the next meeting. It is recommended, therefore, that this standard be continued until the revised procedure is issued.

Standard Method of Test for Permanent Linear Change After Reheating of Refractory Brick (C 113 – 36).—It is recommended that this method be continued as standard without revision.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 38 members, with the results shown in Table I.

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[5] 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 417.

[6] *Ibid.*, p. 419.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARD			
Spec. for Fireclay Plastic Refractories for Boiler Furnaces and Incinerator Service	29	1	3
II. REVISION OF STANDARD, IMMEDIATE ADOPTION			
Testing Insulating Fire Brick (Compressive Strength, Flexural Strength, and Permanent Linear Change After Heating) (C 93 – 41)	32	0	1
III. ADOPTION OF TENTATIVE STANDARD AS STANDARD			
Def. of Terms Relating to Refractories (Abrasion, Burning, Corrosion, Erosion, and Slagging) (C 71 – 40 T)	28	0	3
IV. ADOPTION AS STANDARD OF TENTATIVE REVISIONS OF STANDARDS			
Test for Pyrometric Cone Equivalent of Refractory Materials (C 24 – 35)	30	0	1
Panel Test for Resistance to Thermal and Structural Spalling of Refractory Brick (C 38 – 36)	30	0	1
Panel Test for Resistance to Thermal and Structural Spalling of High Heat Duty Fireclay Brick (C 107 – 40)	30	0	1
Panel Test for Resistance to Thermal and Structural Spalling of Super Duty Fireclay Brick (C 122 – 40)	30	0	1

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Industrial Survey (L. C. Hewitt, chairman) has the first draft prepared on a survey of refractory conditions in electric steel furnaces. The manuscript on refractories in the oil industry is being revised. Arrangements have been made for the preparation of a survey of refractory service in water gas generators.

Subcommittee II on Research (R. S. Bradley, chairman) has been inactive during the past year.

Subcommittee III on Tests (F. A. Harvey, chairman) is composed of the chairmen of the several sections described below. The activities of the subcommittee have already been brought out in the foregoing recommendations on standards and are further amplified in the following discussion of the work of the individual sections:

Section B on Spalling (R. E. Birch, chairman) recommended adoption as standard of tentative revisions of the panel test methods for spalling, Standards C 38 – 36, C 107 – 40, and C 122 – 40.

A Recommended Practice for the Panel Spalling Test has been prepared by the Refractories Fellowship at Mellon Institute in cooperation with the Section on Spalling. This explains in considerable detail items which are not included in the present A.S.T.M. description of the test and has to do with technique, procedure, and equipment. Copies have been sent to five laboratories that use panel spalling furnaces for the purpose of obtaining their suggestions in the hope that eventually the recommended practice will be in an approved form.

Consideration has been given to the suggestion made at the Granville meeting relating to a method for determining thermal shock resistance of silica brick. No time has been available for laboratory study of this subject, and a review of the literature on the subject is being made before attempting to have experimental work carried out.

Section C on Temperature (R. B. Sosman, chairman) has promoted cooperative studies in several research laboratories on the use of gas-fired and electrically heated P.C.E. furnaces.

Section D on Slagging (C. E. Bales, chairman). A review of the literature since the last report was published in 1932 has been prepared by the Refractories Fellowship of Mellon Institute. This will be published in the July, 1942, *Journal* of the American Ceramic Society.

Section E on Analysis (L. J. Trostel, chairman) is continuing its studies on the determination of ferrous oxide in chrome refractories. Mr. G. E. Seil of E. J. Lavino and Co. has furnished the committee with a method used in his laboratory. This is being tried by various members of the section.

Section F on Tests on Refractory Insulation (W. R. Kerr, chairman) recommended the revision of Methods C 93 – 41, discussed earlier in this report.

Section G on Porosity and Permanent Linear Change (C. E. Fulton, chairman) recommended the continuation of Standard Method C 113 – 36, without modification.

Section H on Consistency and Plasticity (P. G. Herold, chairman) is making a review of the literature on the subject of plasticity and related methods of test. This survey was presented by Mr. Herold and Mr. W. Smothers at the April, 1942, meeting of the American Ceramic Society.

Subcommittee IV on Heat Transfer (R. H. Heilman, chairman) in conference with M. S. Van Dusen of the National Bureau of Standards has been advised that the work being done there on a standard apparatus for measuring thermal conductivity of refractory materials has been recessed, possibly for the duration of the war.

Cooperative work has been in progress for some time in the laboratories of the Babcock & Wilcox Co. under the direction of C. L. Norton, Jr., of this committee, and in the Johns-Manville Corp. under the direction of C. B. Bradley, also of our committee. One laboratory used a water-flow calorimeter and the other the conventional heat-flow electrical method. Samples of insulating fire brick have been measured in both apparatus. Refinements in technique and of the apparatus resulted in obtaining checks of unusually good reproducibility. It is believed that the work has progressed to the point where a recommended test method will be presented at the fall 1942 meeting of Committee C-8.

It now appears that both types of equipment will be suitable for determining with good reproducibility, the thermal conductivity of insulating fire brick. The work at the Bureau of Standards was intended to develop a more precise method, but as this seems too far in the future, the subcommittee will proceed at this time on the basis of the work which it has done itself.

Subcommittee V on Precision and Tolerance (R. A. Heindl, chairman) has no report to submit this year.

Subcommittee VI on Nomenclature (M. E. Holmes, chairman) has spent considerable effort in trying to bring the definitions of terms relating to refractories in line with the requirements of Committee E-8 on Nomenclature and Definitions. The recommendations, previously outlined, with respect to Tentative Definitions C 71 – 40 T originated in this subcommittee.

Subcommittee VII on Specifications (G. A. Bole, chairman) presented the proposed Tentative Specification for Fireclay Plastic Refractories for Boiler Furnaces and Incinerator Service, mentioned earlier in this report.

The subcommittee is also preparing specifications on cold-set mortars. A copy of the first draft has been submitted to the members of the subcommittee for discussion and suggestions. It is expected that these specifications will be in shape to present at the fall meeting of Committee C-8 for publication as tentative.

Subcommittee VIII, Editorial (L. J. Trostel, chairman) handled all routine matters and is now considering the feasibility of issuing late in 1942 or early in 1943 a new edition of the Manual of A.S.T.M. Standards on Refractory Materials. The 1941 edition of 2700 copies is exhausted, and the Society does not favor a reissue since many changes in standards have been made since that time. When the 1941 Manual was issued, Committee C-8 had not expected to put out a revised edition until 1944, but in view of the unexpected demand for copies it may be desirable to have an earlier edition.

Subcommittee IX on Classifications (E. H. Van Schoick, chairman) has no report to submit this year.

This report has been submitted to letter ballot of the committee, which consists of 38 members; 36 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. D. Sullivan,
Chairman.
L. J. Trostel,
Vice-Chairman.

S. M. Phelps,
Secretary.

REPORT OF COMMITTEE C-9

ON

CONCRETE AND CONCRETE AGGREGATES

Committee C-9 on Concrete and Concrete Aggregates held four meetings contributing to this report: one in Chicago, Ill., on June 26, 1941; one in Baltimore, Md., on December 6, 1941; one in Cleveland, Ohio, on March 3, 1942; and one during the 1942 annual meeting of the Society at Atlantic City, N. J.

Arthur Newell Talbot, Honorary Chairman of Committee C-9, died on April 3 at the age of 84. Professor Talbot's death represents a great loss to Committee C-9, to the engineering profession, and to the country. Few men have accomplished as much in the advancement of engineering knowledge. He was active to the end.

Arthur Newell Talbot was born on October 21, 1857, at Cortland, Illinois. After graduation from the University of Illinois in 1881, he went west and was for four years engaged in railroad location, construction and maintenance. In 1885 he returned to the University and in 1890 was made Professor of Municipal and Sanitary Engineering, in charge of Theoretical and Applied Mechanics. He continued in this position until September, 1926, when he reached the age limit established by the University, at which time he retired from teaching and administration and was made Professor of Municipal and Sanitary Engineering, *Emeritus*.

Before the turn of the century Professor Talbot had made contributions to the engineering profession in hydrology, railway engineering, sanitary engineering, and testing materials which brought him distinction. In 1903 he started comprehensive investigations of reinforced concrete which were continued for many years. The conception of relations existing between the strength of a concrete mixture and items involving the absolute volumes of the ingredients, submitted in a paper[1] in 1921, stands as a classic on the theory of concrete mixtures. His most recent notable activity, begun in 1914 and continued until his death, involved a study of stresses in railroad track which has contributed greatly to comfort and safety of travel in high-speed trains.

Professor Talbot exercised a far-reaching influence on engineering developments through committee activity in engineering societies. He took a leading part in the work of the first Joint Committee on Concrete and Reinforced Concrete (1904–1916). He was active in the American Society for Testing Materials from its beginning in 1898. He served as President of the Society for the year 1913–1914. He was a member of Committee C-9 since its organization in 1914 and was elected Honorary Chairman in 1936.

Professor Talbot was a member of numerous organizations and his work was recognized by honorary memberships in many of them, including the American Society for Testing Materials, and by numerous medals and awards, including the John Fritz Medal by the United Engineering Societies.

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[1] A. N. Talbot, "A Proposed Method of Estimating the Density and Strength of Concrete and of Proportioning the Materials by the Experimental and Analytical Consideration of the Voids in Mortar and Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 21, p. 940 (1921).

Sanford E. Thompson Award.—The Sanford E. Thompson Award for a paper of outstanding merit on concrete and concrete aggregates presented at an annual meeting of the Society will be made this year to John Tucker, Jr., for his paper on "Statistical Theory of the Effect of Dimensions and of Method of Loading Upon the Modulus of Rupture of Beams,"[2] which was presented at the 1941 annual meeting of the Society. This is the third Sanford E. Thompson Award, previous ones having been made to W. T. Thomson for his paper on "A Method of Measuring Thermal Diffusivity and Conductivity of Stone and Concrete"[3] presented at the 1940 annual meeting and to T. F. Willis and M. E. DeReus for their paper on "Thermal Volume Change and Elasticity of Aggregates and Their Effect on Concrete"[3] presented at the 1939 annual meeting.

Significance of Tests.—The Report on Significance of Tests of Concrete and Concrete Aggregates, first published in 1935, is in the process of being revised under the direction of a special committee of which R. W. Crum is chairman. In most cases the revised chapters are being brought up to date by the original authors. The revised symposium is expected to be ready for publication early this fall and will include the following papers:

Introduction
Compressive Strength of Concrete, by Inge Lyse as revised by R. R. Litehiser
Tensile and Flexural Strengths of Concrete, by A. T. Goldbeck
Shearing and Torsional Strengths of Concrete, by F. E. Richart
Elastic Properties of Concrete, by L. W. Teller
Durability of Concrete, by C. H. Scholer
Wear Resistance of Concrete, by L. H. Tuthill and R. F. Blanks
Workability of Concrete, by T. C. Powers
Volume Changes of Concrete, by R. E. Davis and J. W. Kelly
Cement Content of Hardened Concrete, by H. F. Kriege
Uniformity of Concrete, by R. L. Bertin
Permeability and Absorption of Concrete, by M. O. Withey
Density and Unit Weight of Concrete, by M. A. Swayze
Tests for Curing Concrete, by W. F. Kellermann
Tests for Size, Shape, Surface Texture, and Grading of Aggregates, by H. J. Gilkey
Specific Gravity, Unit Weight and Voids of Aggregates, by Fred Hubbard
Soundness of Aggregates, by Stanton Walker
Abrasion of Aggregates, by H. F. Clemmer
Free Moisture and Absorption of Aggregates, by Bert Myers
Deleterious Substances, by F. C. Lang
Mortar Strength of Fine Aggregate, by F. H. Jackson
Mineral Composition of Aggregates, by H. F. Kriege
The Numbers of Specimens or Tests of Concrete and Concrete Aggregates Required for Reasonable Accuracy of the Average, by R. W. Crum and H. W. Leavitt

Subsequent to the 1941 annual meeting, Committee C-9 presented to the Society through Committee E-10 on Standards the following recommendations:

Revision of Tentative Method of Test for:

Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 88 - 39 T),

Tentative Revisions of Standard:

Specifications for Lightweight Aggregate for Concrete (C 130 - 39),
Method of Making and Storing Compression Test Specimens of Concrete in the Field (C 31 - 39),
Method of Test for Compressive Strength of Concrete (C 39 - 39),
Method of Securing Specimens of Hardened Concrete from the Structure (C 42 - 39),
Method of Test for Compressive Strength of Concrete Using Portions of Beams Broken in Flexure (Modified Cube Method) (C 116 - 39),
Method of Test for Specific Gravity and Absorption of Coarse Aggregate (C 127 - 39), and

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[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 1072 (1941).

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 1073 (1940), and Vol. 39, p. 919 (1939).

Method of Test for Specific Gravity and Absorption of Fine Aggregate (C 128 – 39).

These recommendations were accepted[4] by Committee E-10 on August 25, 1941, and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part II.

I. NEW TENTATIVE STANDARDS

The committee recommends that the following new specifications and methods be accepted for publication as tentative, as appended hereto.[5]

Tentative Specifications for:

Waterproof Paper for Curing Concrete, and

Tentative Methods of:

Sampling Fresh Concrete,
Measuring the Length of Drilled Concrete Cores, and
Test for Air Content of Freshly Mixed Concrete.

II. REVISION OF TENTATIVE STANDARDS

The committee recommends that the Tentative Specifications for Ready Mixed Concrete (C 94 – 41 T)[6] be revised as indicated below and continued as tentative. The changes for the most part are of an editorial nature and reflect suggestions resulting from the publication of these tentative specifications which were issued last year as a tentative revision of the Standard Specifications for Ready Mixed Concrete (C 94 – 38) which latter standard is recommended for withdrawal later in this report.

Section 2.—Change the title of this section from "Definitions" to read "Description of Terms."

Change this section from its present form: namely,

> 2. (*a*) *Ready Mixed Concrete.*—The term ready mixed concrete is used to describe mixed concrete (either central mixed or transit mixed) delivered in a plastic state to the site of the work ready for use.
>
> (*b*) *Central Mixed Concrete.*—The term central mixed concrete is used to describe ready mixed concrete for which the required mixing is done at a central mixing plant.
>
> (*c*) *Shrink Mixed Concrete.*—The term shrink mixed concrete is used to describe ready mixed concrete that has been partially mixed at a central mixing plant and the mixing completed in a truck mixer.
>
> (*d*) *Transit Mixed Concrete.* — The term transit mixed concrete is used to describe ready mixed concrete mixed in a truck mixer.

to read as follows:

> 2. (*a*) *Ready-mixed concrete* is central-mixed concrete or transit-mixed concrete transported and delivered in a plastic and unhardened state ready for placement in the work.
>
> (*b*) *Central-mixed concrete* is ready-mixed concrete for which the required mixing is done at a central-mixing plant.
>
> (*c*) *Shrink-mixed concrete* is ready-mixed concrete that has been partially mixed at a central-mixing plant and the mixing completed in the truck mixer.
>
> (*d*) *Transit-mixed concrete* is ready-mixed concrete mixed in a truck mixer.

Section 12 (*d*).—Change to read as follows by the omission of the words in brackets:

> (*d*) The concrete shall be considered as conforming to the requirements of these specifications if the average strength of the specimens cut from the structure is equal to or greater than the specified strength [and at least 90 per cent of all tests are equal to or greater than 90 per cent of the specified strength].

Footnote 6.—Indicate the paragraph marked with an asterisk as Paragraph (*d*), relettering the present Paragraph (*d*) as (*e*).

Footnote 7.—Omit the last sentence which reads as follows:

> At least 90 per cent of all strength tests should be equal to or greater than 90 per cent of the strength specified.

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[4] In submitting these recommendations to Committee E-10 on Standards, Committee C-9 reported the following results of the letter ballot vote of 61 ballots returned from a membership of 74: C 88, affirmative 50, negative 1, ballots marked "not voting" 10; C 130, affirmative 57, negative 1, ballots marked "not voting" 3; C 31, C 39, C 42, and C 116, affirmative 60, negative 0, ballots marked "not voting" 1; C 127, affirmative 58, negative 2, ballots marked "not voting" 1; C 128, affirmative 59, negative 1, ballots marked "not voting" 1.

[5] These specifications and methods were accepted as tentative by the Society and appear in the 1942 Book of A.S.T.M. Standards, Part II, pp. 1214, 1202, 1207, 1203, respectively.

[6] 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 321.

Insert a new Footnote 8 to refer to Section 10 (*a*) and Section 12 (*d*) to read as follows and renumber the subsequent footnotes accordingly:

[8] It is desirable that at least 90 per cent of all strength tests be equal to or greater than 90 per cent of the strength specified. However, attention is directed to the fact that such a requirement is impracticably rigid unless a considerable number of specimens are involved—probably not less than about 20.

The committee recommends that the Tentative Definitions of Terms Relating to Concrete and Concrete Aggregates (C 125 – 36 T)[7] be revised by the inclusion of new definitions for "flat piece," "elongated piece," "water-cement ratio," and a revised definition of "blast furnace slag." The new and revised definitions are as follows:

Flat Piece.—In the case of concrete aggregate, one in which the ratio of the width to thickness of its circumscribing rectangular prism is greater than a specified value.

Elongated Piece.—In the case of concrete aggregate, one in which the ratio of the length to width of its circumscribing rectangular prism is greater than a specified value.

Water Cement Ratio.—The ratio of the amount of water, exclusive only of that absorbed by the aggregates, to the amount of cement in a concrete mixture. This ratio is variously stated as follows: (1) by bulk volume of cement (assuming cement to weigh 94 lb. per cu. ft.); (2) by absolute volume of cement; (3) by weight; and (4) in terms of gallons of water per 94 lb. sack of cement.

Blast Furnace Slag.—The nonmetallic product consisting essentially of silicates and aluminosilicates of lime, and of other bases, which is developed simultaneously with iron in a blast furnace.

III. REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption, revisions in the following standards and accordingly asks for a favorable nine-tenths vote at the annual meeting in order that these modifications may be referred to letter ballot of the Society:

Standard Specifications for Concrete Aggregates (**C 33 – 40**):[8]

Section 3 (*a*).—Change the maximum permissible limit on material finer than a No. 200 sieve for aggregate used in concrete subject to surface abrasion from "3 per cent" to read "4 per cent."

The above change in Section 3 (*a*) and those recommended for inclusion in the specification with the adoption of the tentative revision of Section 4 (*a*) of Specifications C 33 – 40 have been made to bring this A.S.T.M. standard in line with what appears to be a well defined trend in requirements for fine aggregate, dictated, in a large measure, by use of leaner mixtures and more finely ground cements. The revision corresponds with action taken by other specification writing bodies such as the Federal Specifications Executive Committee and the American Association of State Highway Officials.

Standard Method of Making and Storing Compression Test Specimens of Concrete in the Field (**C 31 – 39**):[9]

Section 5.—Change from its present form to read as follows:

> 5. Samples of concrete for test specimens shall be taken in accordance with the Tentative Method of Sampling Fresh Concrete (A.S.T.M. Designation: C 172) of the American Society for Testing Materials.[10] The location in the work of the batch of concrete thus sampled shall be noted for further reference.

Standard Method of Test for Cement Content of Hardened Portland-Cement Concrete (**C 85 – 36**):[11]

Section 4 (*b*).—Change the last sentence of this section to read as follows by the addition of the italicized words and the omission of those in brackets:

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[7] 1939 Book of A.S.T.M. Standards, Part II, p. 996.

[8] 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 84.

[9] 1939 Book of A.S.T.M. Standards, Part II, p. 324.

[10] 1942 Book of A.S.T.M. Standards, Part II, p. 1202.

[11] 1939 Book of A.S.T.M. Standards, Part II, p. 322.

> Determine the silica present in the sample by treatment with HF and H_2SO_4 [or by fusion with $NaHSO_4$] *in accordance with the procedure given in Section 6 of the Standard Methods of Chemical Analysis of Portland Cement (A.S.T.M. Designation: C 114 – 40) of the American Society for Testing Materials.*

Section 4 (c).—Renumber as Section 5 with the title "Correction Factors" and change to read as follows by the addition of the italicized word; also renumber the present Section 5 as 6:

> 5. When the aggregates are available which were used in the concrete being analyzed, a blank test shall be run on these aggregates to determine their content of silica, soluble under the conditions of the *above* test. This content of soluble silica shall then be used as a correction factor and be subtracted from the total soluble silica found in the concrete, the difference being due to the cement contained in the specimen.

Standard Method of Test for Unit Weight of Aggregate (**C 29 – 39**):[12]

Section 2 (c).—In the table of dimensional requirements for the cylindrical measure, change the minimum thickness of metal, U. S. gage from "No. 11" to indicate thickness of metal, U. S. gage as "No. 10 to No. 12" for all three capacities.

VI. ADOPTION AS STANDARD OF TENTATIVE REVISIONS OF STANDARDS

The committee recommends that the tentative revision[13] submitted in June, 1941 in the Standard Specifications for Concrete Aggregates (C 33 – 40)[8] which provides for a change in Section 4 (*a*) be approved for reference to letter ballot of the Society for adoption as standard. The 1941 revision as well as Section 4 (*a*) of Standard C 33 – 40, requires the fine aggregate to be so graded that from 5 to 30 per cent shall pass a No. 50 sieve and from 0 to 8 per cent shall pass a No. 100 sieve. It is now recommended that these limitations be changed to read as follows:

Sieve	Percentage Passing
No. 50 (297-micron)	10 to 30
No. 100 (149-micron)	2 to 10

It is further recommended that the following qualifying notes be added immediately after the table of fine aggregate grading requirements, renumbering the present note as Note 3:

> NOTE 1.—At the approval of the Engineer, when the fine aggregate is to be used in concrete mixtures containing five (5) or more bags of cement per cubic yard, the limitations on the No. 50 and No. 100 sieves may be 5 to 30 and 0 to 10 per cent, respectively.
>
> NOTE 2.—Fine aggregate failing to pass the minimum requirements for the material passing the No. 50 and/or No. 100 sieve may be used provided a satisfactory inorganic fine material is added to correct for the difference in grading.

The committee recommends that the tentative revision[14] submitted in June, 1940 in the Standard Method of Test for Compressive Strength of Concrete (C 39 – 39)[15] which provides for a change in Section 15 pertaining to storage of test specimens be approved for reference to letter ballot of the Society for adoption as standard with a minor change in the second sentence substituting the words "free water" for "free moisture."

The committee also recommends that the tentative revisions of the following seven specifications and methods be approved for reference to letter ballot of the Society for adoption as standard:

Standard Specifications for:

Lightweight Aggregates for Concrete (C 130 – 39),[16]

Standard Methods of:

Making and Storing Compression Test Specimens of Concrete in the Field (C 31 – 39),[16]

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[12] 1939 Book of A.S.T.M. Standards, Part II, p. 308.

[13] 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 420.

[14] *Ibid.*, p. 424.

[15] 1939 Book of A.S.T.M. Standards, Part II, p. 328.

[16] 1939 Book of A.S.T.M. Standards, Part II, pp. 285, 324, 320, 304, 333, 300, 302.

Securing Specimens of Hardened Concrete from the Structure (C 42 – 39),[16]
Test for Structural Strength of Fine Aggregate Using Constant Water-Cement Ratio Mortar (C 87 – 39),[16]
Test for Compressive Strength of Concrete Using Portions of Beams Broken in Flexure (Modified Cube Method) (C 116 – 39),[16]
Test for Specific Gravity and Absorption of Coarse Aggregate (C 127 – 39),[16] and
Test for Specific Gravity and Absorption of Fine Aggregate (C 128 – 39).[16]

V. WITHDRAWAL OF STANDARDS

The committee recommends that the Standard Specifications for Ready-Mixed Concrete (C 94 – 38)[17] be withdrawn and replaced by the Tentative Specifications C 94 – 41 T, revised as indicated earlier in the report.

The committee also recommends that the Standard Method of Test for Voids in Inundated Fine Aggregate (C 69 – 30)[17] be withdrawn because it no longer represents a standard applicable in practice for the testing of concrete aggregates.

VI. TENTATIVE STANDARDS CONTINUED AS TENTATIVE

The committee recommends that the following methods of test be continued as tentative:

Tentative Methods of Test for:

Soundness of Aggregates by Freezing and Thawing (C 137 – 38 T),
Efficiency of Materials for Curing Concrete (C 156 – 40 T), and
Volume Change for Cement Mortar and Concrete (C 157 – 40 T).

VII. EDITORIAL CHANGE IN STANDARD

The following footnote reference in connection with Section 12 on Molds for Test Specimens in the Standard Method of Test for Compressive Strength of Concrete (C 39–39) is recommended as an editorial change:

For a more complete description of the mold reference may be made to Section 3 of the Standard Method of Making and Storing Compression Test Specimens of Concrete in the Field (A.S.T.M. Designation: C 31) of the American Society for Testing Materials.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 72 members with the results shown in Table I.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee V, Editorial and Definitions (C. M. Chapman, chairman).—This subcommittee is giving consideration to the formulation of other definitions applying to concrete and concrete aggregate including such terms as "curing," "silt," "crushed gravel," and "permeability."

Subcommittee VI on Relation of Characteristics of Materials and Mixtures to Properties of Concrete (H. J. Gilkey, chairman).—The scope of work of this subcommittee is sufficiently general to permit it to undertake almost any problem within the field of Committee C-9 which is not readily classified. An important assignment before it at the present time has to do with a study of chemical reactions between aggregates and alkalies.

Subcommittee VII on Methods and Apparatus for Testing Concrete (A. T. Goldbeck, chairman).—This subcommittee has been quite active as is indicated by the three new methods completed this year and the several recommendations and revisions in existing methods as mentioned earlier in this report.

The new Proposed Tentative Method of Sampling Fresh Concrete was prepared as a result of a request that a standard sampling procedure for concrete be formulated. While certain of the present test methods contain brief descriptions of a sampling pro-

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[17] 1939 Book of A.S.T.M. Standards, Part II, pp. 314, 311.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Spec. for Waterproof Paper for Curing Concrete	43	1	13
Sampling Fresh Concrete	54	1	2
Measuring Length of Drilled Concrete Cores	52	0	5
Test for Air Content of Freshly Mixed Concrete	46	3	9
II. REVISIONS OF TENTATIVE STANDARDS			
Spec. for Ready Mixed Concrete (C 94 – 41 T)	49	0	1
Def. of Terms Relating to Concrete and Concrete Aggregates (C 125 – 36 T):			
Flat piece	53	4	2
Elongated piece	54	3	2
Water cement ratio	59	0	0
Blast furnace slag	57	0	2
III. REVISIONS OF STANDARDS, IMMEDIATE ADOPTION			
Spec. for Concrete Aggregates (C 33 – 40)	42	4	4
Making and Storing Compression Test Specimens of Concrete in the Field (C 31 – 39)	50	0	0
Test for Cement Content of Hardened Portland-Cement Concrete (C 85 – 36)	47	1	9
Test for Unit Weight of Aggregate (C 29 – 39)	53	0	3
IV. ADOPTION AS STANDARD OF TENTATIVE REVISION OF STANDARD			
Spec. for Concrete Aggregates (C 33 – 40)	49	1	0
Spec. for Lightweight Aggregates for Concrete (C 130 – 39)	43	0	7
Making and Storing Compression Test Specimens of Concrete in the Field (C 31 – 39)	50	0	0
Test for Compressive Strength of Concrete (C 39 – 39)	50	0	0
Securing Specimens of Hardened Concrete from the Structure (C 42 – 39)	50	0	0
Test for Structural Strength of Fine Aggregate Using Constant Water-Cement Ratio Mortar (C 87 – 39)	48	0	2
Test for Compressive Strength of Concrete Using Portions of Beams Broken in Flexure (Modified Cube Method) (C 116 – 39)	45	1	3
Test for Specific Gravity and Absorption of Fine Aggregate (C 127 – 39)	47	0	3
Test for Specific Gravity and Absorption of Fine Aggregate (C 128 – 39)	47	0	3
V. WITHDRAWAL OF STANDARDS			
Spec. for Ready Mixed Concrete (C 94 – 38)	49	0	1
Test for Voids in Inundated Fine Aggregate (C 69 – 30)	53	0	4

cedure there has been a need for a more detailed method. The procuring of a proper sample has a very important bearing on test results and it seemed highly desirable that the method of sampling should be described in sufficient detail to insure that a representative sample is obtained in an adequate manner.

This subcommittee also completed the Proposed Tentative Method of Test for Air Content of Fresh Concrete. The measurement of air contained in fresh concrete is an important problem because the air content affects the volume of concrete produced with a given quantity of the respective materials. Certain grinding aids in the production of portland cement seem to produce a large volume of void space in the concrete and when these cements are used, that void space becomes a very significant property of the concrete and, hence, a method for determining it becomes essential.

In support of the Proposed Tentative Method of Test for Length of Drilled Concrete Cores there is presented a project report prepared by L. W. Teller which appears in the Appendix.

It now has before it for consideration a proposed procedure for measuring the relative rate of bleeding, or water gain, of freshly mixed concrete. It also has on its agenda the development of a specification for rate of loading of concrete test specimens.

Subcommittee VIII on Permeability of

Concrete.—A method of test for measurement of permeability of concrete is being prepared by this subcommittee.

Subcommittee IX on Specifications and Methods of Tests of Aggregates (H. F. Clemmer, chairman).—This subcommittee is now considering a complete revision of the Standard Method of Test for Surface Moisture in Fine Aggregate (C 70 – 30). The principal purpose of the revision is to provide more flexibility in the type of equipment used to conduct the test. Other projects are the development of specifications for fine aggregates resulting from the crushing of stone, slag, or gravel and the development of a suitable test procedure for identifying soft particles in aggregates.

Subcommittee XI on Evaluation of Data (H. W. Leavitt, Chairman).—Studies of number of samples and test results required to furnish reliable data are being continued.

Subcommittee XIII on Curing of Concrete (W. F. Kellermann, chairman).—This subcommittee prepared the Proposed Tentative Specifications for Waterproof Paper for Curing Portland Cement Concrete mentioned earlier in this report. Waterproof paper has been used for curing for a number of years without complete specifications or tests to determine its suitability for this purpose having been standardized. It was for this reason that the subcommittee undertook the preparation of these specifications which provide requirements for waterproof paper having the desired properties for curing concrete. These proposed specifications have been reviewed and approved by Committee D-6 on Paper and Paper Products.

Subcommittee XIV on Workability of Concrete (F. R. McMillan, chairman).—Studies are being made of the workability of concrete and methods for measurement of that property.

Subcommittee XVI on Elasticity and Volume Changes of Concrete (L. W. Teller, chairman).—This subcommittee has been developing further information on volume changes of concrete.

Subcommittee XVII on Conditions Affecting Durability of Concrete in Structures (C. H. Scholer, chairman).—A review has been made of recent work relating to durability of concrete as well as an investigation of the causes of concrete failures.

Subcommittee XVIII on Ready-Mixed Concrete. (R. B. Young, Chairman).—This subcommittee is continuing as its principal activity a careful scrutiny of specifications for ready-mixed concrete and their utility in controlling the quality of that product. It is now engaged in developing a measure of adequate uniformity of acceptance tests of ready mixed concrete.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, F. H. Jackson.
Vice-Chairman, F. E. Richart.
Secretary, Stanton Walker.

This report has been submitted to letter ballot of the committee which consists of 72 members; 56 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

F. H. JACKSON,
Chairman.

STANTON WALKER,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee C-9 recommended to Committee E-10 on Standards that the following A.S.T.M. standards relating to curing be withdrawn:

Standard Specifications for:
Curing Portland-Cement Concrete (C 80 – 34),

Standard Methods of:
Curing Portland-Cement Concrete Slabs with Bituminous Coverings (C 81 – 36),
Curing Portland-Cement Concrete with Calcium Chloride Admixture (C 82 – 38),
Curing Portland-Cement Concrete Slabs by Surface Application of Calcium Chloride (C 83 – 38), and
Curing Portland-Cement Concrete Slabs with Wet Coverings (C 84 – 36).

This recommendation was accepted by Committee E-10 on August 24, 1942, and the standards have accordingly been discontinued.

PROJECT REPORT ON A STUDY OF METHODS OF MEASUREMENT OF THE LENGTH OF CORES DRILLED FROM CONCRETE STRUCTURES

REPORT OF SUBCOMMITTEE VII ON METHODS AND APPARATUS FOR TESTING CONCRETE

PREPARED BY L. W. TELLER[1]

A number of years ago Committee C-9 on Concrete and Concrete Aggregates undertook the development of a method by which the length of cores drilled from concrete structures could be determined. The determination of core length is important when the measurement is used to determine whether or not a concrete slab or other structure has been built to the specified depth or thickness. It is common practice, for example, to test the depth of completed concrete pavement slabs by measuring the length of cores drilled from them. Since under some specifications the contractor may be subjected to penalties if the slab is found to be deficient in thickness, it is apparent that the measurement obtained from the core must truly represent the thickness of the slab or structural member from which the core was drilled.

On first thought it might appear that the problem is very simple; that the length of a right cylinder taken from a plate or slab could have but one length, which would necessarily be the thickness of the plate. Practically, however, the ends of a core are seldom, if ever, smooth planes at right angles to the axis of the cylinder. In the case of a concrete pavement, the lower surface of the slab is not perfectly smooth because it is cast in contact with moist earth which deforms as the fresh concrete is deposited and manipulated, while the upper surface may be deliberately roughened to provide a skid-resistant texture. Furthermore, a core drilled from a concrete pavement may have its lower end altered by spalling, which frequently occurs as the bit breaks through the pavement slab at the completion of the drilling operation. Any portion of the end area which is not part of the original surface of the slab obviously should be avoided in making length measurements. Thus, the ends of the core may not be smooth, may not be plane, and may contain areas that are not usable for the purpose of measurement.

It must be recognized also that, as in any other length determination, no two measurements will give exactly the same result. An essential step in the development of the method of measurement is, therefore, a decision as to the degree of precision that is necessary or desirable in the length determination. Once this tolerance of measurement is established, the problem is one of finding a method that will determine in a fair and equitable manner the distance between the original surfaces which formed the core ends and that will at the same time consistently remain within the tolerance.

Survey of State Highway Methods:

As a first step in the study, a questionnaire was sent to 24 highway departments asking for a description of the

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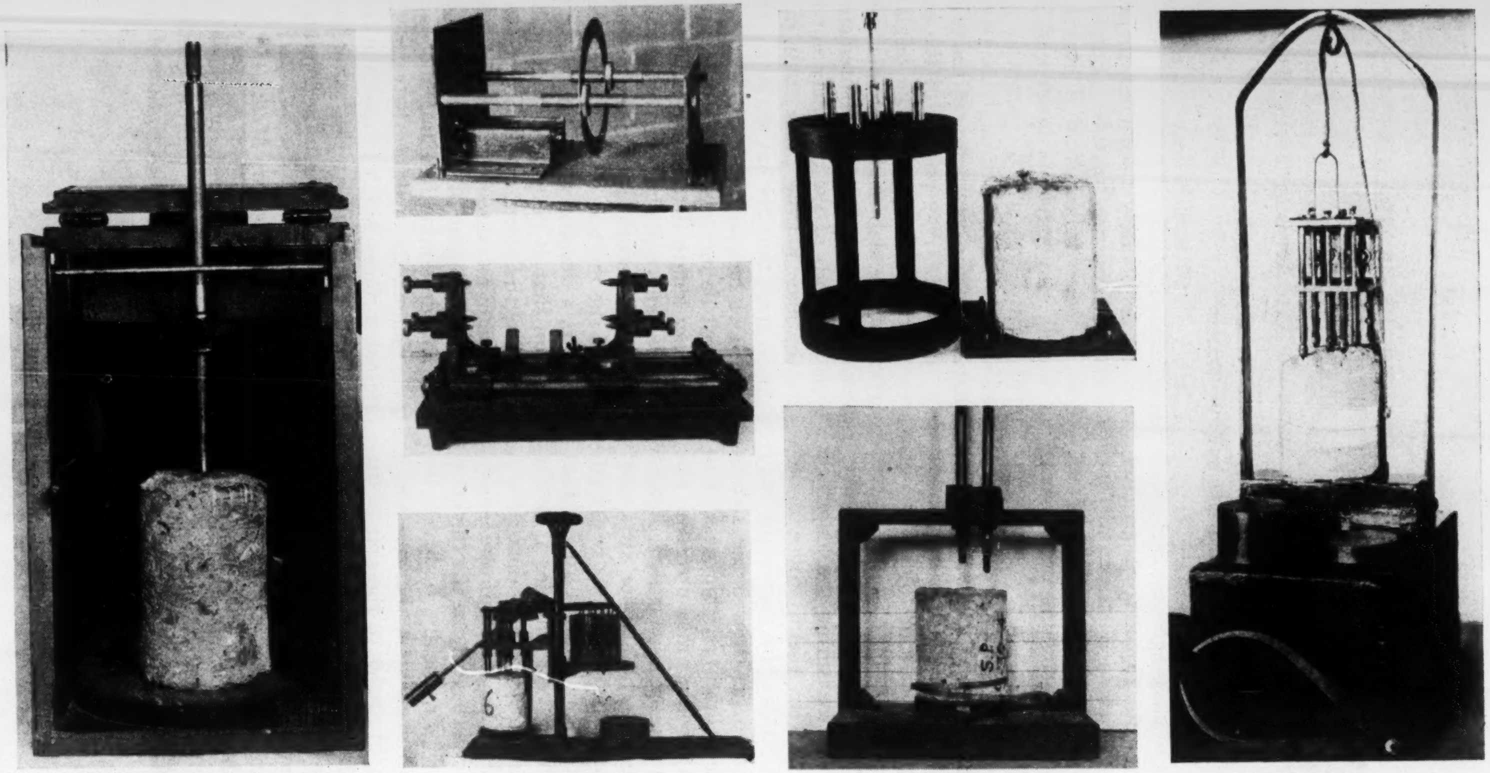
[1] Senior Engineer of Tests, Public Roads Administration, Federal Works Agency, Washington, D. C.



Pennsylvania

Iowa
North Carolina
South Carolina

Michigan
Minnesota

Missouri

FIG. 1—Several Designs of Core Measuring Apparatus in Current Use by State Highway Departments.

method which each used for determining core length and an estimate of the accuracy of length determination obtained by the method. The cooperation obtained from these departments was very gratifying and from the information received certain significant generalizations may be drawn, as follows:

1. The measuring method that has been generally adopted is a direct calipering procedure of some sort.
2. The number of points on the core end used in the length determination varies from 1 to 10 or more.
3. The type of apparatus varies widely in mechanical design. (Several designs illustrating the range in methods used by state highway departments are shown in Fig. 1.)
4. The estimates of accuracy of length determination varied from 0.01 to 0.125 in.

Laboratory Studies:

In addition to the survey of the measuring methods being used by state highway laboratories, certain studies were undertaken in the laboratory of the Public Roads Administration. It seemed desirable to investigate rather thoroughly the possibilities of direct calipering methods, particularly to determine how the indicated length of a core might be affected by the number and position of the points selected for measurement.

For this laboratory study a special measuring apparatus was designed. This device consists of a base, in the center of which is a rotatable specimen table that can be locked in any one of eight positions 45 deg. apart, a rigid but adjustable frame supported by the base and establishing a measuring datum at a known distance above the specimen, and a graduated rod with a rounded tip which can be placed at any one of nine positions with respect to the axis of the core and which measures the distance from the datum plate to the end surface of the core.

The specimen is actually supported by three rounded steel studs fixed in the specimen table. The overhead datum plate can be raised or lowered and locked into grooves placed 1 in. apart on the supporting columns to accommodate specimens of various lengths. The nine holes in the datum plate are arranged in a simple spiral pattern and provide a center position and eight others on radii 45 deg. apart and separated by equal radial increments of $\frac{5}{16}$ in., making the outermost position $2\frac{1}{2}$ in. from the center or axis of rotation of the core. The measuring rod is graduated in tenths of inches and in use is read to hundredths by estimation. The details of the device are shown in Fig. 2. Before any cores were measured, the various possibilities for error in the device itself were investigated and, where necessary, correction factors were determined, to be applied later in the core length measurements.

The specimens used for the study comprised approximately 100 cores that happened to be available in the laboratory at the time. These cores had been drilled from pavements in Ohio and Texas and included various mixes, pavement depths, methods of consolidation, etc. The general appearance of a representative group of the specimens is shown in Fig. 3. It is believed that these specimens as a whole are fairly typical of those obtained generally from concrete pavements with the shot type of core drill.

Each core was placed on the three rounded supporting studs on the table of the measuring device, with the pave-

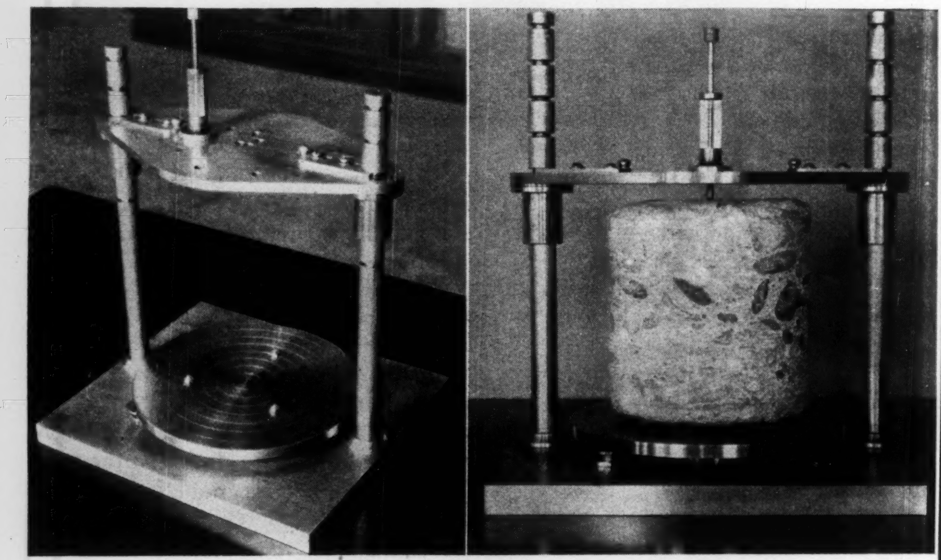


FIG. 2.—Core Measuring Apparatus Used in the Investigation.

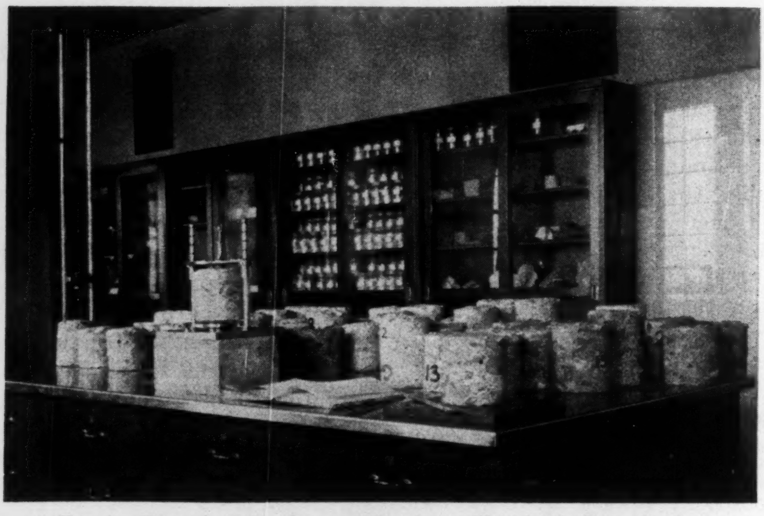


FIG. 3.—Representative Group of Specimens Used in the Investigation.

ment-surface end down. Its position was adjusted until the upper end of the core was centered under the central hole in the datum plate.

Nine measurements were made with the graduated measuring rod, one measurement for each hole or position in the datum plate. The core was then rotated in 45-deg. increments, and for each of the eight increments nine measurements were made with the graduated rod. For each core, 72 observations were obtained by this procedure, eight of which were repetitions of the mid-point or central point measurement. While, theoretically, the eight repetitions of the measurement at the central position should give identical values, actually this is not always the case. In Table I the maximum variation in the eight length observations at the central position for each of 129 sets of core measurements are summarized.

TABLE I.—MAXIMUM VARIATION IN EIGHT REPETITIONS OF MID-POINT LENGTH MEASUREMENTS.

Maximum Variation, in.	Number of Specimens Having Indicated Maximum Variation
0.00	15
0.01	65
0.02	33
0.03	7
0.04	5
0.05	1
0.06	2
0.11	1

It is apparent that in most cases the variation was quite small, yet occasionally some local abnormality existed in the central region that caused an appreciable variation in the indicated core length as the core was revolved through the eight positions. Usually this was observed to be a projecting bit of aggregate or an irregular depression. It should be noted that the maximum variation values in this table apply only to the eight presumably identical measurements at the center of one specimen and do not indicate the accuracy of the mid-point measurement as related to the real length of the core.

Analysis of Data:

Having obtained the complete length measurement data on 102 cores in the manner described above, it was possible to compare the indicated core lengths for a range of numbers of measurements and for a variety of measuring point patterns and it seemed that the relation between numbers of measurements and accuracy of length determination could best be developed by statistical analysis methods. The matter was discussed with H. W. Leavitt, chairman of Subcommittee XI on Evaluation of Data, who volunteered to undertake the analysis as a subcommittee project. The work has been completed and the analysis has been described in some detail in a report by Subcommittee XI.[2]

The data obtained with the core measuring device may be considered as observations in a series of nine concentric circles with radii varying in increments of $\frac{5}{16}$ in. from 0 to $2\frac{1}{2}$ in.

It was mentioned earlier that when the core drill bit breaks through, at the conclusion of the drilling operation, some spalling around the edges of the lower end of the core is likely to occur. In the measuring procedure described above, the core is inverted and the spalled edges are up. For this reason, measurements near the circumference of the end area are open to question. In the analysis of the data, comparisons were made between the variations within the several concentric circles of measurement and the variation among the means of these circles of measurement, it being reasonable to assume that the variation between circles should not be significantly different from the variation within circles. When this criterion was applied to the data it was found that, in the case of the Ohio cores, the measurements from the five inner circles and, in the case of the Texas cores, those from the four inner circles were within the tolerance.

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[2] Presented as a discussion of this report, see p. 256.—ED.

This significant result indicates that, in general, when measuring cores of this size (nominal diameter 6 in.) the points of measurement should be confined to a central area having a diameter of about 2½ in. This suggests that the requirements for a method of measurement should delimit the area in which the length measurements are made, preferably by subtracting from the actual radius of the specimen some constant that would eliminate the area affected by spalling and using the remainder as the radius of the area in which the measurements may be made.

TABLE II.[a]—RELATION BETWEEN ACCURACY AND NUMBER OF MEASUREMENTS FOR VARIOUS ALLOWABLE TOLERANCES (BASED ON COMBINED DATA FROM OHIO AND TEXAS CORES, INNER FOUR CIRCLES ONLY).

Tolerance, in.	Accuracy, per cent, Obtained When				
	$N^b = 5$	$N^b = 9$	$N^b = 17$	$N^b = 25$	$N^b = 33$
0.10.....	99.97	100.00	100.00	100.00	100.00
0.08.....	99.63	99.99	100.00	100.00	100.00
0.06.....	97.04	99.65	100.00	100.00	100.00
0.05.....	93.01	98.50	99.92	100.00	100.00
0.04.....	85.30	94.83	99.25	99.88	99.98
0.03.....	72.32	85.54	95.51	98.50	99.46
0.02.....	53.16	66.93	81.87	89.50	93.75
0.01.....	28.30	37.32	49.61	58.24	64.83

[a] This appears as Table VI in the analysis by Subcommittee XI, see p. 259.

[b] N is the number of measurements.

The analysis then proceeds to develop a very useful relation between the tolerance in length determination, number of measurements, and percentage of accuracy. The accompanying Table II, taken from the report of the analysis by Subcommittee XI, shows this interrelationship.

If a decision can be made as to a satisfactory tolerance in length determinations, it is apparent that the data in this table make it possible to reach some conclusion regarding the number of measurements that should be made on an individual core.

In the survey of core measurement practice mentioned previously, the estimates of accuracy obtained by the various agencies varied from 0.01 to 0.125 in. A number of laboratories indicated that an accuracy of 0.05 in. was considered satisfactory. This would seem to be a reasonable tolerance value to consider as it would mean an accuracy of length determination of better than 1 per cent for cores of usual length.

If the tolerance of 0.05 in. can be accepted, the data in Table II show that should nine measurements be made within the prescribed area on a given core end, it can be assumed that the accuracy of length determination will be within this tolerance 98.5 per cent of the time. Nine points of measurement is a rather desirable number since it lends itself to a very simple geometric pattern, a central position with eight others 45 deg. apart arranged around a circle, a pattern that is already in use by several state highway departments.

Reproducibility of Results:

In order to obtain some indication of the reproducibility that might be expected from successive measurements on a given core when employing the 9-point pattern suggested by the analysis, certain supplemental measurements have been made.

For this work ten typical concrete pavement cores were selected. Using the 72-point pattern described earlier, each specimen was measured 10 times. Each specimen was also measured 10 times using the suggested 9-point pattern. When making these measurements, the specimen was lifted from its support in the measuring apparatus, rotated slightly (about 4½ deg.) and placed back on the supports between each of the measurements in the series of 10, in order that a different set of points on the core end would be used for each length determination.

From the supplemental measurements just described the data shown in Table III were obtained.

It will be noted that when the 9-point measuring pattern is used, the maximum spread between the least indicated length and the greatest indicated length is 0.05 in. Comparing the average length as obtained with the 9-point pattern and that with the 72-point pattern, it is interesting to observe that wherever a significant difference exists, the length as determined by the 9-point measurement is greater than that determined by the 72-point measurement. The 72-point pattern includes a number of points near the periphery of the specimen and the measurements at these points are affected by the presence of spalling around the periphery. On cores that are spalled, therefore, measurements which are confined to the undamaged central portion of the core end will indicate a somewhat greater average core length and a length that is a more dependable measure of the real thickness of the slab from which the core was drilled.

TABLE III.—REPRODUCIBILITY DATA FROM TEN COMPLETE LENGTH DETERMINATIONS ON EACH SPECIMEN.

Core	Core Length, in.			
	72-Point Pattern	9-Point Pattern		
	Avg.	Min.	Max.	Avg.
No. 1	6.71	6.70	6.73	6.71
No. 2	5.64	5.64	5.67	5.66
No. 3	5.77	5.79	5.80	5.79
No. 4	5.85	5.84	5.89	5.86
No. 5	5.88	5.87	5.92	5.90
No. 6	5.70	5.67	5.70	5.69
No. 7	6.03	6.06	6.06	6.06
No. 8	7.78	7.81	7.84	7.83
No. 9	8.77	8.79	8.83	8.80
No. 10	6.26	6.29	6.32	6.31

Summary:

To summarize the results of the study it is indicated that:

1. A direct calipering of core lengths is the method of measurement preferred by most agencies.
2. An accuracy of length determination of 0.05 in. is reasonably satisfactory for the length determination for cores of usual lengths.
3. To insure this degree of accuracy in 98.5 per cent of the cases, each specimen length must be determined by averaging not less than nine well-distributed individual measurements.
4. The area in which these individual measurements are made must be limited to the central part of the core end. The area generated by rotating the outer 1½ in. of the radius of the end area of the core apparently includes that portion of the core end which should be excluded.

With these indications as a basis a Method of Measuring the Length of Drilled Concrete Cores[3] has been prepared and is recommended by Subcommittee VII for submission to the Society for publication as tentative.

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[3] This method was accepted as tentative by the Society and appears in the 1942 Book of A.S.T.M. Standards, Part II, p. 1207.

ANALYSIS OF DATA ON LENGTH OF CONCRETE CORES

Discussion of Report of Subcommittee VII by Subcommittee XI on Evaluation of Data

Under date of June 21, 1941, a preliminary report showing the results of initial studies of Ohio and Texas core data was submitted by Subcommittee XI, of Committee C-9, to L. W. Teller. Curves indicating the numbers of measurements theoretically necessary for various degrees of accuracy were presented. Charts showing possible locations for symmetrically arranged, representative points to be measured were also included.

After consideration of the preliminary report, Mr. Teller indicated his choices for locations of points to be measured from the various groupings submitted, in some instances selecting different groupings from those suggested in the report. Because the concrete cores appeared somewhat rounded or chipped off near the edge of the core, it was Mr. Teller's opinion that the outer concentric circle of points measured (see Fig. 1) should not be included in determining the true length of the core.

This suggestion that the outer ring of measurements be discarded was the immediate reason for the analysis of the data given in this report. If the outer circle of measurements should not be included in determining the true length of a core, might it not be true that the next to the outer circle should also be thrown out? Perhaps even a third circle should be eliminated.

In order to determine at which points the measurements were valid, the methods of the analysis of variance were applied, first to the Ohio data (41 cores), second to the Texas data (61 cores), and then to the combined data (102 cores).

The method consisted in comparing the variations within the several concentric circles of measurements of each core with the variations among the means of these circles of measurements. If all of the measurements in the end area are sufficiently uniform, the variation between circles would not be significantly different from the variation within circles. In case the outer one or two circles gave measurements considerably different from those of the inner circles, the variation between means of circles would be found significantly different from the variation within circles. Thus the analysis of variance offers a means of determining the validity of the measurements of the outer circles of points.

The Ohio Core Data

The 41 Ohio cores were studied first. When all eight concentric circles were considered, the variation between means of circles was found to be significantly greater than the variation within the circles of measurements. This meant that the outside or eighth (from the center) circle should not be included, thus substantiating Mr. Teller's opinion.

The data for the remaining seven circles were then similarly analyzed. Again it was found the variation between circles was significantly greater (although to a lesser degree) than the variation within circles. The seventh circle, therefore, should also be discarded.

Application of the method to the remaining six circles of measurements, again gave a similar result, although this time the difference between variation within circles and variation between circles was so slight as to be on the border between *significant* and *not significant*. However, as the 5 per cent point had not been reached, the rejection of the sixth circle was indicated.

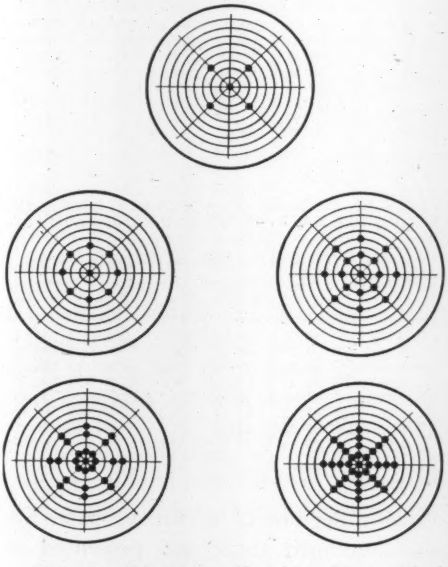

FIG. 1.—Diagrams Showing Symmetrical Arrangements of Measurement Points for Five Values of N Indicated in Tables IV, V, and VI.

The data for the remaining five inner circles were then analyzed; the results indicating that the measurements in these five circles were all valid.

This variance analysis showed that the three outer circles of measurements of the Ohio cores should be discarded and the measurements confined to the 41 (including the center) points in the area covered by the five inner circles.

For a number of measurements, the standard deviation of the mean of those measurements is found by the formula:

$$\sigma_m = \frac{\sigma}{\sqrt{N}}$$

For the Ohio data the standard deviation of the measurements included in the five inner circles was found to be 0.055557 in. The standard deviation of the mean, then, varies inversely with the square root of the number of points measured. Statistical tables show the percentage of cases included by various numbers of standard deviations measured plus and minus from the means. The number of measurements required to obtain a certain accuracy may be found by means of the formula:

$$x\,\sigma_m = T$$

where

x = number of standard deviations of the mean,

σ_m = standard deviation of the mean,

σ = standard deviation of the individuals, and

T = allowable tolerance or variation from true mean length.

From this formula it is found that:

$$N = \left(\frac{x\sigma}{T}\right)^2$$

Table I indicates the numbers of measurements necessary, based on the

TABLE I.—NUMBER OF MEASUREMENTS BASED ON OHIO CORE DATA—INNER FIVE CIRCLES ONLY.

Allowable Tolerance, in.	Number of Measurements for Accuracy of		
	68.26%[a]	95.46%[a]	99.73%[a]
0.10	0.31	1.23	2.78
0.08	0.48	1.93	4.34
0.06	0.86	3.43	7.72
0.05	1.23	4.94	11.11
0.04	1.93	7.72	17.36
0.03	3.43	13.72	30.87
0.02	7.72	30.87	69.45[b]
0.01	30.87	123.46[b]	277.79[b]

[a] Included by one, two, and three standard deviations, respectively.

[b] Outside of limits of these data.

Ohio data, for various degrees of accuracy.

THE TEXAS CORE DATA

The analysis of variance methods were next applied to the data for the 61 Texas cores. The results were similar to those of the Ohio cores. In this case it was found necessary to discard an additional circle of measurements, as only the four inner circles of measurements were found to yield valid results. Thus only 33 points (including the center) could be used in determining the lengths of these Texas cores.

TABLE II.—NUMBER OF MEASUREMENTS BASED ON TEXAS CORE DATA—INNER FOUR CIRCLES ONLY.

Allowable Tolerance, in.	Number of Measurements for Accuracy of		
	68.26%[a]	95.46%[a]	99.73%[a]
0.10	0.46	1.83	4.11
0.08	0.71	2.85	6.42
0.06	1.27	5.07	11.41
0.05	1.83	7.30	16.43
0.04	2.85	11.41	25.68
0.03	5.07	20.29	45.64[b]
0.02	11.41	45.64[b]	102.70[b]
0.01	45.64[b]	182.58[b]	410.80[b]

[a] Included by one, two, and three standard deviations, respectively.

[b] Outside of limits of these data.

TABLE III.—NUMBER OF MEASUREMENTS BASED ON COMBINED OHIO AND TEXAS CORE DATA—INNER FOUR CIRCLES ONLY.

Allowable Tolerance, in.	Number of Measurements for Accuracy of		
	68.26%[a]	95.46%[a]	99.73%[a]
0.10	0.38	1.52	3.42
0.08	0.59	2.38	5.35
0.06	1.06	4.23	9.51
0.05	1.52	6.09	13.70
0.04	2.38	9.51	21.40
0.03	4.23	16.91	38.05[b]
0.02	9.51	38.05[b]	85.61[b]
0.01	38.05[b]	152.20[b]	342.45[b]

[a] Included by one, two, and three standard deviations, respectively.

[b] Outside of limits of these data.

Table II indicates the numbers of measurements necessary, based on the Texas data, for various degrees of accuracy.

COMBINED OHIO AND TEXAS CORE DATA

When the Ohio and Texas data were combined the results were the same as in the case of the Texas cores alone. Only the four inner circles of measurements yield valid results.

The numbers of measurements for various degrees of accuracy, based on the combined data, are presented in Table III.

TABLE IV.—DEGREE OF ACCURACY BASED ON OHIO CORE DATA—INNER FIVE CIRCLES ONLY.

Allowable Tolerance, in.	Accuracy, per cent, Obtained When				
	$N = 5$	$N = 9$	$N = 17$	$N = 25$	$N = 33$
0.10	100.00	100.00	100.00	100.00	100.00
0.08	99.87	100.00	100.00	100.00	100.00
0.06	98.43	99.88	100.00	100.00	100.00
0.05	95.58	99.31	99.98	100.00	100.00
0.04	89.26	96.92	99.70	99.97	100.00
0.03	77.28	89.48	97.40	99.31	99.81
0.02	57.92	71.98	86.22	92.81	96.14
0.01	31.27	41.08	54.20	63.19	69.89

TABLE V.—DEGREE OF ACCURACY BASED ON TEXAS CORE DATA—INNER FOUR CIRCLES ONLY.

Allowable Tolerance, in.	Accuracy, per cent, Obtained When				
	$N = 5$	$N = 9$	$N = 17$	$N = 25$	$N = 33$
0.10	99.91	100.00	100.00	100.00	100.00
0.08	99.19	99.96	100.00	100.00	100.00
0.06	95.40	99.23	99.97	100.00	100.00
0.05	90.21	97.36	99.77	99.98	100.00
0.04	81.45	92.43	98.54	99.69	99.95
0.03	67.93	81.72	93.29	97.36	98.93
0.02	49.20	62.55	77.78	86.12	91.10
0.01	25.94	34.02	45.84	54.08	60.48

The results shown in Tables I, II, and III are shown in different arrangement in Tables IV, V, and VI, respectively. These tables indicate the degree of accuracy obtained by different numbers of measurements for various allowable tolerances.

The data presented in these six tables show that the number of measurements necessary to determine the true length of a core depends upon two factors; first, the allowable tolerance or variation from the true mean; second, the number of times out of 100 when it is considered necessary to come within that tolerance.

The numbers heading the columns in Tables IV, V, and VI were selected because it is possible to select such numbers of symmetrically arranged points within the area included by the four inner circles of measurements. The accompanying diagrams, shown in Fig. 1, illustrate such symmetrically arranged numbers of points.

TABLE VI.—DEGREE OF ACCURACY BASED ON COMBINED OHIO AND TEXAS CORE DATA—INNER FOUR CIRCLES ONLY.

Allowable Tolerance, in.	Accuracy, per cent, Obtained When				
	$N = 5$	$N = 9$	$N = 17$	$N = 25$	$N = 33$
0.10.....	99.97	100.00	100.00	100.00	100.00
0.08.....	99.63	99.99	100.00	100.00	100.00
0.06.....	97.04	99.65	100.00	100.00	100.00
0.05.....	93.01	98.50	99.92	100.00	100.00
0.04.....	85.30	94.83	99.25	99.88	99.98
0.03.....	72.32	85.54	95.51	98.50	99.46
0.02.....	53.16	66.93	81.87	89.50	93.75
0.01.....	28.30	37.32	49.61	58.24	64.83

It has been shown that the Ohio cores as a group differ from the Texas cores as a group. It is thought that the combined data for the two groups offer a better basis for conclusions than either group singly, since a method for determining core length should be generally applicable. Table VI, therefore, offers a basis for some interesting observations.

For example, if a tolerance of 0.05 in. is allowable, the average of nine measurements of core length would lie within that amount of the true length 98.50 per cent of the time. If 17 measurements were made the average would be within 0.05 in. of the true core length 99.92 per cent of the time, etc. It will be noted also that the average of nine measurements would lie within 0.03 in. of the true length only 85.54 per cent of the time, and within 0.01 in. only 37.32 per cent of the time.

CONCLUSION

In order to establish a certain number of measurements as being the required number, it is, therefore, necessary to fix arbitrarily the allowable tolerance and the desired percentage of accuracy.

Mr. Teller suggested 0.05 in. as a suitable tolerance limit. It would seem that an accuracy of 98.50 per cent would be sufficient for all practical purposes. For these criteria, nine symmetrical, representative measurements made within the area included by the inner four circles should produce the required accuracy.

REPORT OF COMMITTEE C-11
ON
GYPSUM

Committee C-11 on Gypsum held one meeting during the past year: on March 5, 1942 in Cleveland, Ohio, in conjunction with the spring group committee meetings of the Society.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, L. S. Wells.
Vice-Chairman, C. K. Roos.
Secretary, H. J. Schweim.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Gypsum Plasters (G. W. Farrell, chairman).—In view of certain questions raised when the A.S. T.M. Specifications for Calcined Gypsum for Dental Plasters (C 72 – 40) were submitted to the American Standards Association for approval as American standard, this subcommittee will conduct during the coming year whatever tests are necessary to establish the relationship between the tensile strength and compressive strength of these plasters. This study will be undertaken with a view to revising Section 4 of the Specifications for Calcined Gypsum for Dental Plasters (C 72 – 40)[1] by substituting requirements for compressive strength in place of those for tensile strength, or by including requirements for both compressive and tensile strength, or by the incorporation of a footnote setting forth the relationship that is considered to exist between the tensile and compressive strengths.

This year the subcommittee is recommending a tentative revision in Section 8 of Standard Specifications C 72 – 40 and also in Section 16 (*b*) of the Standard Methods of Testing Gypsum and Gypsum Products (C 26 – 40),[2] as given later in this report.

Subcommittee III on Structural Gypsum Products (L. S. Wells, chairman).—This subcommittee has reviewed the Standard Specifications for Gypsum Lath (C 37 – 40),[3] Gypsum Wall Board (C 36 – 34),[4] and Gypsum Sheathing Board (C 79 – 34),[5] and recommends a number of changes in these specifications to bring them up to date.

Subcommittee IV on Testing Methods (C. K. Roos, chairman).—This subcommittee has been working for the past several years on the ammonium acetate method for use as an alternate method for determining the purity of gypsum.[6] The proposed method was published last year as an appendix to the report of Committee C-11. During the year work on this problem has been continued and the subcommittee now recommends its inclusion as an alternate method in Standard Methods C 26.

During the coming year the committee will study a proposed method for determining the water permeability of gypsum sheathing board and as a long-time project will continue its study of subsieve fineness determination of gypsum particles.

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[1] 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 41.
[2] *Ibid.*, p. 55.
[3] *Ibid.*, p. 53.
[4] 1939 Book of A.S.T.M. Standards, Part II, p. 84.
[5] *Ibid.*, p. 82.
[6] *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 293 (1941).

I. TENTATIVE REVISION OF STANDARDS

In the Standard Specifications for Calcined Gypsum for Dental Plasters (C 72 – 40),[1] Section 8 on methods of testing contains an exception from the Standard Methods of Testing Gypsum and Gypsum Products (C 26 – 40).[2] The committee felt that for uniformity of methods of testing this exception should be avoided. With the deletion of the requirement concerning the consistency at which the time of set and tensile strength should be determined and the deletion of the reference to the soaking period, a note should be added to Section 16 (*b*) of Standard Methods C 26 relative to the use of retarder when determining the testing consistency of quick setting calcined gypsum. Committee C-11 accordingly presents for publication as tentative the following revisions of Standard Specifications C 72 and Standard Methods C 26:

Standard Specifications for Calcined Gypsum for Dental Plasters (C 72 – 40):[1]

Section 8.—Change to read as follows by the omission of the words in brackets:

> 8. The chemical analysis and physical properties of calcined gypsum for use in the preparation of dental plasters shall be determined in accordance with the Standard Methods of Testing Gypsum and Gypsum Products (A.S.T.M. Designation: C 26) of the American Society for Testing Materials. [Except that in the determination of the time of setting and the tensile strength the calcined gypsum and water shall be mixed in the proportion of 100 g. of calcined gypsum to 60 ml. of water. The calcined gypsum shall be allowed to soak in distilled water at 70 F. (21 C.) for 30 sec., and shall then be stirred for 30 sec. and placed immediately in the molds.]

Standard Methods of Testing Gypsum and Gypsum Products (C 26 – 40):[2]

Section 16 (*b*).—Add a note to read as follows:

> NOTE.—The use of 0.1 per cent retarder in determining the testing consistency is necessary when working with quick-setting calcined gypsum products.

II. REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

Standard Specifications for Gypsum Lath (C 37 – 40).[3]

A careful study of Standard Specifications C 37 – 40, C 36 – 34, and C 79 – 34 indicates the advisability of a number of revisions to bring them up to date. In addition to plain and perforated gypsum lath referred to in Section 1 on scope there is another type of mechanical key lath that should be provided for. On the Pacific Coast $\frac{5}{16}$-in. gypsum lath is commonly used. Requirements, therefore, for this thickness of lath should be included. The note following Section 6 (*b*) makes reference only to aluminum foil. This material is at present on the priority list; the industry, therefore, is making use of a different type of foil. The committee recommends for immediate adoption the following revisions in Standard Specifications C 37 and accordingly asks for a favorable nine-tenths vote at the annual meeting in order that this change may be referred to letter ballot of the Society:

Section 1.—Change from its present form: namely,

> 1. These specifications cover both plain and perforated gypsum lath, gypsum plaster boards designed to be used as a base for the reception of gypsum plaster.

to read as follows:

> 1. These specifications cover plain, perforated, and other mechanical key types of gypsum lath which are designed to be used as a base for the reception of gypsum plaster.

Section 3.—Change the table of breaking loads to read as follows by the addition of the italicized figures:

Thickness in.	Load, lb. Knife Edges Across Fiber of Surfacing	 Knife Edges Parallel to Fiber of Surfacing
¼	40	16
5/16	*52*	*22*
⅜	60	27
½	100	40

Section 5 (*a*).—Change to read as follows by the addition of the italicized word and figure:

> (*a*) *Thickness.*—The nominal thickness of gypsum lath shall be ¼ in., *5/16 in.*, ⅜ in., or ½ in., with permissible variations in the nominal thickness of plus or minus $\frac{1}{32}$ in., and with permissible local variations of plus or minus $\frac{1}{16}$ in. from the nominal thickness.

Section 5 (*d*).—Change the table on weight of gypsum lath to read as follows by the addition of the italicized figures:

Thickness, in.	Weight per 1000 sq. ft., lb. Minimum	 Maximum
¼	900	1500
5/16	*1125*	*1750*
⅜	1350	2000
½	1800	3000

Section 6 (*b*).—Change the note to read as follows by the addition of the italicized words:

> NOTE.—Gypsum lath is also available with one surface covered with aluminum *or other heat-reflecting type of* foil.

Standard Specifications for Gypsum Wall Board (C 36 – 34):[4]

In addition to the widths, lengths, and type edges referred to in these specifications, gypsum wall board is also available in 24-in. widths, 14 ft. lengths, with either plain or recessed edges, or edges and ends, and with either square or V-tongue and groove edges. The manufacture of beveled edge board has been discontinued. Committee C-11 recommends for immediate adoption the following revisions in Standard Specifications C 36 and accordingly asks for a favorable nine-tenths vote at the annual meeting in order that these changes may be referred to letter ballot of the Society.

Section 5 (*a*).—Add the following sentence at the end of this section:

> Edges, or edges and ends of ⅜ in. and ½ in. board may be plain or recessed to receive a joint reinforcing strip; if recessed, the depression shall be not less than 0.02 in.

Section 5 (*b*).—Change to read as follows by the addition of the italicized word and figure and the omission of the words in brackets:

> (*b*) *Width.*—The nominal width of gypsum wall board shall be *24 in.*, 32 in., 36 in., or 48 in., with a permissible variation of $\frac{3}{32}$ in. under the specified width. [Beveled edge wall boards may have widths ¼ in. less than those specified, with the same permissible variation.]

Section 5 (*c*).—Change from its present form: namely,

> (*c*) *Length.*—The nominal length of gypsum wall board shall be from 4 ft. to 12 ft., inclusive, with permissible variations of plus or minus ¼ in. from the specified length.

to read as follows:

> (*c*) *Length.*—The nominal length of ¼-in. gypsum wall board shall be from 4 ft. to 12 ft. and of ⅜-in. and ½-in. board from 4 ft. to 14 ft., inclusive, with permissible variations of plus or minus ¼ in. from the specified length.

Section 6.—Add a new note as Note 1 to read as follows:

> NOTE 1.—Gypsum wall board in ½-in. thickness is available either with square edges or V-tongue and groove edges. Recessed board is available only with square edges.

Designate the present note as Note 2 and change from its present form to read as follows:

> NOTE 2.—Gypsum wall board is also available with one surface covered with aluminum or other heat-reflecting type of foil.

Standard Specifications for Gypsum Sheathing Board (C 79 – 34):[5]

Gypsum sheathing board is not available in 32 in. width nor in ¾ in. thickness. The practice of applying aluminum or other heat-reflecting foils to gypsum sheathing board has been discontinued. Gypsum sheathing board is available either with V-tongue and groove edges or square edges. The committee therefore recommends for immediate adoption the following revisions in Standard Specifications C 79 and accordingly asks for a favorable nine-tenths vote at the annual meeting in order that these changes may be referred to letter ballot of the Society.

Section 3.—Omit the requirements for flexural strength of $\frac{3}{4}$-in. board which now specify a load of 175 lb. when tested across fiber of surfacing and 100 lb. parallel to fiber of surfacing.

Section 5.—In Paragraph (*a*) eliminate reference to $\frac{3}{4}$-in. board in the first sentence.

In Paragraph (*b*) change to read as follows by the omission of the words and figures in brackets:

(*b*) *Width.*—The nominal width of gypsum sheathing board shall be [either] 24 in. [or 32 in.], with a permissible variation of plus or minus $\frac{1}{8}$ in. from the specified width.

In Paragraph (*d*) omit weight requirements for $\frac{3}{4}$-in. board now specified as minimum of 3000 lb. and maximum of 4000 lb. per 1000 sq. ft.

Section 6.—Change the note following this section from its present form: namely,

NOTE.—Gypsum sheathing board is also available with one or both surfaces covered with aluminum foil.

to read as follows:

NOTE.—Gypsum sheathing board is available either with square edges or V-tongue and groove edges.

Standard Methods of Testing Gypsum and Gypsum Products (**C 26 – 40**):[2]

Included in the 1941 report of Committee C-11 was a recommendation for the deletion of the test for absorption of gypsum partition tile or block described in Sections 37 and 38, which change the committee had originally proposed become effective immediately. As the change was a minor one and the standard involved is twelve pages long the Committee on Papers and Publications suggested that the change become effective in 1942 when the new Book of Standards is issued rather than in 1941. Committee C-11 gladly acquiesced in that request. The committee accordingly recommends that the deletion of Sections 37 and 38 from Methods C 26 now be approved for reference to letter ballot of the Society.

For the past few years several members of Subcommittee IV have been conducting tests comparing the ammonium acetate method with the gravimetric method for determining the purity of gypsum and calcined gypsum and last year the proposed ammonium acetate method was published as an Appendix to the report of Committee C-11. It is felt that sufficient investigative work has been done on this method to justify its inclusion as an alternate procedure in Standard Methods C 26. The committee therefore recommends this alternate analytical method[7] for immediate adoption as a revision of Standard Methods C 26 and accordingly asks for a favorable nine-tenths vote at the annual meeting in order that this recommendation may be referred to letter ballot of the Society.

III. ADOPTION OF TENTATIVE STANDARD AS STANDARD

Tentative Definitions of Terms Relating to Gypsum (**C 11 – 41 T**):[8]

The present tentative definition of perforated gypsum lath states that the perforations in all perforated gypsum lath are not less than $\frac{3}{4}$-in. in diameter and would require a minimum number of perforations. That this is not necessary is self evident. However, to obtain a

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[7] The new alternate method was adopted as standard by the Society and incorporated in the Standard Methods of Testing Gypsum and Gypsum Products (C 26 – 42), which appear in their latest form in the 1942 Book of A.S.T.M. Standards, Part II, p. 116.

[8] 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 266.

1-hr. fire rating, which is required by most building codes for certain locations, this number and size of perforations are necessary. Therefore, the committee recommends that the present definition of perforated gypsum lath be revised to read as follows by the addition of the italicized words and that it be submitted to Committee E-8 with the recommendation that it be approved for presentation to the Society for adoption as standard:[9]

Perforated Gypsum Lath.—A gypsum lath with perforations. *For one hour fire resistive construction, perforated gypsum lath shall be not less than ⅜ in. in thickness with perforations* not less than ¾ in. in diameter with one perforation for not more than each 16 sq. in. of lath.

IV. WITHDRAWAL OF TENTATIVE REVISION OF STANDARD

Standard Specifications for Gypsum (**C 22–41**):[10]

This tentative revision[11] which proposed increasing the $CaSO_4 \cdot 2H_2O$ content of gypsum from 64.5 to 75 per cent was commented on quite freely to the effect that inasmuch as raw gypsum for use as a retarder in portland cement is purchased on the SO_3 content and that finished products are required to comply with the specifications for that particular product, there was no need to increase the present $CaSO_4 \cdot 2H_2O$ content. The committee, therefore, recommends that this tentative revision be withdrawn.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 22 members; 21 members returned their ballots, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. TENTATIVE REVISIONS OF STANDARDS			
Spec. for Calcined Gypsum for Dental Plasters (C 72 – 40; ASA A65.1–1941)	19	0	2
Tests for Gypsum and Gypsum Products (C 26 – 40; A70.1–1941)	21	0	0
II. REVISIONS OF STANDARDS			
Spec. for Gypsum Lath (C 37 – 40; A67.1–1941), immediate adoption	21	0	0
Spec. for Gypsum Wall Board (C 36 – 34; A69.1–1941), immediate adoption	20	1	0
Spec. for Gypsum Sheathing Board (C 79 – 34; A68.1–1941), immediate adoption	20	1	0
Tests for Gypsum and Gypsum Products (C 26 – 40; A70.1–1941), immediate adoption	21	0	0
III. ADOPTION OF TENTATIVE STANDARD AS STANDARD			
Def. of Terms Relating to Gypsum (C 11 – 41 T)[9]	20	1	0
IV. WITHDRAWAL OF TENTATIVE REVISION OF STANDARD			
Spec. for Gypsum (C 22 – 41; A49.1–1941)	20	1	0

This report has been submitted to letter ballot of the committee which consists of 22 members; 21 members returned their ballots, of whom 20 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. S. WELLS,
Chairman.

H. J. SCHWEIM,
Secretary.

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[9] The recommendation for adoption as standard of this revised tentative definition failed of approval at the annual meeting of the Society, see Summary of Proceedings, p. 31.

[10] 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 5.

[11] 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 337.

REPORT OF COMMITTEE C-12

ON

MORTARS FOR UNIT MASONRY

Committee C-12 on Mortars for Unit Masonry has held three meetings during the past year: in Chicago, Ill., on June 25, 1941, in Washington, D. C., on December 5, 1941, and in Cleveland, Ohio, on March 3, 1942.

The membership at present totals 34, of whom 13 are classified as producers, 7 as consumers, and 14 as general interest members.

REVISION OF TENTATIVE STANDARD

The committee also recommends that the Tentative Specifications for Aggregate for Masonry Mortar (C 144 – 39 T) be revised as appended hereto[1] and continued as tentative. The revision will add clearly defined limits on gradation, which were not included heretofore.

This recommendation has been submitted to letter ballot of the committee which consists of 34 members; 20 members returned their ballots, of whom 17 have voted affirmatively, 0 negatively, and 3 members marked their ballots "not voting."

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Research (F. O. Anderegg, chairman) has continued its investigations on properties of mortars and development of new test methods. A report on the results of a series of cooperative tests made under the direction of this subcommittee has been prepared and appears in the Appendix to this report.

Subcommittee III on Specifications and Methods of Test for Mortar (R. B. Crepps, chairman) has submitted to Committee C-12 proposed Tentative Specifications for Mortar for Concrete Masonry Units which are under consideration in the main committee. These specifications were prepared by a working subcommittee whose report was referred to Subcommittee III for review and recommendation.

Subcommittee IV on Specifications for Aggregate for Mortar (Stanton Walker, chairman) prepared the extensive revision of the Tentative Specifications for Aggregate for Masonry Mortar (C 144-39 T). The revised specifications are appended hereto.[1]

Subcommittee V on Admixtures for Mortar (W. C. Voss, chairman) has been considering the general principles involved in specifications for admixtures. No recommendations have been offered.

This report has been submitted to letter ballot of the committee which consists of 34 members; 24 members returned their ballots, of whom 22 have voted affirmatively and 2 negatively.

Respectfully submitted on behalf of the committee,

J. W. McBurney,
Chairman.

H. C. Plummer,
Secretary.

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[1] These revised specifications were accepted as tentative by the Society and appear in their latest form in the 1942 Book of A.S.T.M. Standards, Part II, p. 1105.

APPENDIX

RESULTS OF COOPERATIVE TESTS ON MASONRY MORTARS

Introduction:

The data presented in this report were provided by a group of cooperating laboratories in response to a request that masonry cement mortars and cement-lime-sand mortars be tested by certain methods. The program was intended to provide answers to the following questions:

1. What is the range in compressive strength of masonry cement mortars where the proportioning is 1:3 by volume?
2. What is the range and probable minimum compressive strength of 1:1:6 cement-lime-sand mortars proportioned by volume?
3. What is the effect on the compressive strength of mortar of varying initial flow?
4. What is the range in ratio of 7-day to 28-day compressive strength of mortar?

Answers to these questions (and others) were obviously necessary if Committee C-12 on Mortars for Unit Masonry was intelligently to formulate specifications for mortars in terms of properties. Correspondence between the officers of Committee C-12 and certain of the cooperating laboratories provided explanations of certain peculiarities in the results and emphasized the necessity of clarifying certain of the test procedures. This additional information is presented as part of the discussion of the numerical data.

All cooperating laboratories were asked to report 7-day and 28-day compressive strengths of mortar cubes made with initial flows of 70, 105, and 135 per cent. The mortars were made from portland cement, lime, and sand in the proportions of 94:40:450 by weight (1:1:6 by volume), and from masonry cement in the proportion of the sack weight of the cement to 225 lb. (1:3 by volume) of dry sand. The A.S.T.M. Tentative Method of Test for Compressive Strength of Portland-Cement Mortars (C 109 – 37 T)[1] was followed. The laboratories used commercial sands and reported their sieve analyses as follows:

Laboratory	Percentages Passing Sieves								
	⅜-in.	No. 4 (4760-Micron)	No. 8 (2380-Micron)	No. 16 (1190-Micron)	No. 20 (840-Micron)	No. 30 (590-Micron)	No. 50 (297-Micron)	No. 100 (149-Micron)	Fineness Modulus
A 1	100	100	98.6	92.6	....	73.2	31.1	6.1	1.98
A 2	100	100	97.6	86.7	....	62.1	23.4	3.9	2.26
B	100	100	98.5	85.7	....	66.2	33.3	4.9	2.11
C	100	99.6	92.8	....	69.4	....	21.6	2.8	2.14
D	100	99.8	96.9	88.9	....	59.5	15.9	2.2	2.37
E	100	100	99.6	97.0	....	81.0	23.0	1.0	1.98
F	100	100	99.4	95.2	....	82.2	21.8	1.2	2.00
G	100	100	100	88.0	....	56.0	14.0	2.5	2.40
H	100	100	99.8	98.4	....	84.6	27.0	6.1	1.84
I	100	100	98.6	90.4	....	68.5	27.4	0.3	2.15
J	100	100	93.0	83.0	....	63.0	24.0	4.0	2.33
K 1	100	100	100	92.5	....	60.5	8.0	1.0	2.38
K 2	100	100	99.5	95.5	....	77.5	31.0	9.0	1.88
N	100	100	99.4	81.8	76.8	64.8	34.0	5.0	2.16

	No. 10 (2000-Micron)	No. 20 (840-Micron)	No. 30 (590-Micron)	No. 40 (420-Micron)	No. 80 (177-Micron)	No. 100 (149-Micron)	No. 200 (74-Micron)
M 1	96.5	68.5	40.0	30.0	6.0	....	4.0
M 3	99.8	95.0	77.0	53.7	18.0	12.8	6.9

Water retentivity measurements expressed as percentages of the initial flow were also requested for all mortars.

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[1] 1939 Book of A.S.T.M. Standards, Part II, p. 892.

Strength of Masonry Cement Mortars:

Committee C-12 has under consideration Proposed Tentative Specifications for Mortar for Concrete Unit Masonry which offer three types of mortar, types A, B, and C, classified by average compressive strength at 28 days of not less than 2500, 600, and 250 psi., respectively. Of the 22 tests on masonry cement mortars with initial flows of 105 per cent, reported in Table I, 17 classify as type B (exceeding 600 psi.) and 5 classify as type C (between 250 and 600 psi.).

The wide range of ratios of 7-day to 28-day compressive strengths will be noted. In general, the masonry cement

TABLE I.—COMPRESSIVE STRENGTHS OF MASONRY MORTARS.

Laboratory	Initial Flow 70 Per cent			Initial Flow 105 Per Cent			Initial Flow 135 Per Cent		
	Compressive Strength, Psi.		Ratio of 7 to 28 Day Compressive Strength, Per Cent	Compressive Strength, Psi.		Ratio of 7 to 28 Day Compressive Strength, Per Cent	Compressive Strength, Psi.		Ratio of 7 to 28 Day Compressive Strength, Per Cent
	7 Days	28 Days		7 Days	28 Days		7 Days	28 Days	
MASONRY CEMENT MORTARS (1:3)									
A	1100	1600	68	870	1300	67	520	800	65
B	640	855	78	700	875	80	585	670	87
C	1420	1940	78	1140	1510	75	930	1110	84
D	810	1420	57	550	1050	52	400	900	44
E 1[a]	215	455	47	170	440	39	115	390	29
E 2[a]	605	1350	45	570	1275	45	500	1060	47
F	2000	2470	81	1720	2280	75	1320	1810	73
I	305	510	60	250	400	68	150	225	67
J 1[a]	515	915	63	420	815	52	285	630	45
J 2[a]	395	740	52	315	630	50	245	435	56
K 1[b]	500	1345	37	265	885	30	195	775	25
K 2[b]	550	1445	38	305	1020	30	210	860	26
L1	910	1125	81	520	710	73	455	685	66
M 1[a]	595	1095	54	310	665	47	275	485	57
M 2[a]	1045	1750	60	550	1100	50	500	800	62
M 3[a]	585	1035	57	355	710	50	245	410	60
M 4[a]	800	1500	53	535	1100	49	375	700	54
M 5[c]	....	....	..	185	360	51	....	....	..
M 6[c]	....	....	..	375	610	62	....	....	..
M 7[c]	....	....	..	210	400	53	....	....	..
M 8[c]	....	....	..	325	525	62	....	....	..
N	2350	2915	81	1815	2375	76	1460	1890	77
Avg.	850	1360	61	565	955	56	485	815	57
Max.	2350	2915	81	1815	2375	76	1460	1890	77
Min.	215	455	37	170	360	30	115	390	25
CEMENT-LIME-SAND MORTARS (1:1:6)									
A	1880	2480	76	1520	2390	64	1360	2310	59
B	390	555	70	295	420	70	225	335	69
C	730	1000	73	520	710	73	320	570	56
D	3050	3850	79	2600	3500	74	2250	2875	78
F	1590	2180	73	1320	1810	73	1090	1540	71
G 1[d]	1800	2765	65	1650	2465	67	1210	1910	64
G 2[d]	1745	2660	66	1465	2520	58	1335	2010	68
H 1[e]	650	1095	60	500	835	60	295	545	54
H 2	1135	1690	67	1010	1475	68	970	1440	67
I	935	1975	48	875	1675	52	740	1500	49
L1	950	1200	79	720	885	81	515	785	66
M 1[f]	2395	4000	60	1960	2875	68	1660	1785	93
M 2[f]	....	....	..	800	1360	59	....	....	..
N	2310	2900	80	2215	2870	77	2000	2715	74
Avg.	1505	2180	69	1245	1840	68	1075	1565	67
Max.	3050	4000	79	2600	3500	81	2250	2875	78
Min.	390	555	48	295	420	52	225	335	49

[a] Different masonry cements.
[b] Same masonry cement. Different sands.
[c] Different sands.
[d] Different limes.
[e] Lime putty instead of hydrate.
[f] Different sands.

mortars with high 7-day strengths show a relatively small increase in strength at 28 days compared with those having low 7-day strengths, as for example, a comparison of mortars A, C, F, and N with E and K, will show. The exception provided by mortar B will be discussed in the section on flow of mortars. Ratios of 7-day to 28-day compressive strengths are in general rather little affected by variation in initial flow. The trend is for increased flow to lower the ratio but in a number of mortars this is reversed.

The only exception to the generalization that compressive strength of mortars decreases as initial flow increases is provided by mortar B at a flow of 105 per cent compared with the same mortar at a flow of 70 per cent. The effect on compressive strength of variations in initial flow showed wide ranges for different masonry cement mortars. A proposal considered by Committee C-12 that "when the sample represents job-mixed mortar, there shall be deducted from the compressive strength requirement 1 per cent for every 1 per cent of flow by which the flow exceeds 80 per cent, except that no deduction shall exceed 50 per cent" appears more liberal than may be necessary since the average strength at 7-days for a flow of 135 per cent is 55 per cent of that reported for a 70 per cent flow. In other words, an increase in flow of 65 per cent lowered the average compressive strength only 45 per cent. The corresponding lowering for 28-day strength is 40 per cent. It will be noted that, in general, change in initial flow affects 28-day strengths less than 7-day strengths. As with the ratio of 7-day to 28-day strength the different masonry cement mortars provide a number of exceptions to this statement.

Strength of Cement-Lime-Sand Mortars: The 1:1:6 cement-lime-sand mortars also present a wide range in compressive strengths. With the exception of mortar H1, all mortars reported in Table I represent mixtures of portland cement and dry hydrate. In mortar H1 a lime putty was used of such proportions that the 94:40 proportion by weight of portland cement to hydrated lime was provided. Except for the comparison of dry hydrate with putty, these data apparently do not indicate that any consistent differences in strength are attributable to differences in materials.

Additional data supplied by laboratory H indicate that for the particular limes used (comparison of a nonplastic mason's hydrate with a highly plastic lime putty) the hydrates give higher strengths. The 28-day strengths at flows of 105 per cent are as follows:

Proportions (by volume)	Compressive Strength at 28 days, psi. With Lime Putty	With Hydrate
1:2:9	225	570
1:1:6	750	1280
1:0.2:3	2800	3460

Laboratory L provided tests on three 1:1:6 mortars made with lime putty. Unfortunately, the initial flows were not stated. The 7-day compressive strengths of these mortars compared with the strength of the 1:1:6 mortar (mortar L1, Table II) using hydrate and having an initial flow of 135 per cent is as follows:

1:1:6 mortars	Compressive Strength at 7 days, psi.
Hydrate	515
Lime putty L6	400
Lime putty L2	325
Lime putty L3	280

Data on flow after suction for these mortars (H and L) are presented as part of the discussion on that subject.

With the exception of mortar B, all of the 1:1:6 mortars met the 600 psi. proposed strength requirement for type B. The ratios of 7-day to 28-day compressive strength are relatively uniform compared with the masonry cement mortars reported in Table I and fit a 2:3 ratio rather well.

TABLE II.—EFFECT OF INITIAL FLOW ON COMPRESSIVE STRENGTHS OF MASONRY MORTARS.

Laboratory	7 Days			28 Days		
	Compressive Strength at 70 per cent Flow, psi.	Percentage Compressive Strength[a] Developed at		Compressive Strength at 70 per cent Flow, psi.	Percentage Compressive Strength[a] Developed at	
		105 per cent Flow	135 per cent Flow		105 per cent Flow	135 per cent Flow
MASONRY CEMENT MORTARS (1:3)						
A	1100	79	47	1600	72	50
B	650	107	90	855	102	78
C	1420	80	65	1940	78	57
D	810	58	49	1420	74	63
E 1[b]	215	79	53	455	97	86
E 2[b]	605	94	83	1360	94	78
F	2000	86	66	2470	92	73
I	305	82	49	510	78	44
J 1[b]	515	81	55	915	89	69
J 2[b]	395	80	62	740	85	59
K 1[c]	500	53	38	1345	66	58
K 2[c]	550	56	38	1445	71	60
L1	910	57	50	1125	69	57
M 1[b]	595	52	46	1095	61	44
M 2[b]	1045	53	48	1750	63	46
M 3[b]	585	61	42	1036	69	40
M 4[b]	800	67	47	1500	73	47
N	2350	77	62	2915	82	65
Avg.	....	72	55	....	79	60
Max.	....	107	90	....	102	86
Min.	....	52	38	....	61	40
CEMENT-LIME-SAND MORTARS (1:1:6)						
A	1880	81	72	2480	96	93
B	390	76	58	555	76	60
C	730	71	44	1000	71	57
D	3050	85	74	3850	91	75
F	1590	83	69	2180	83	71
G 1[d]	1800	92	67	2765	89	69
G 2[d]	1745	84	76	2660	95	76
H 1[e]	650	77	46	1095	76	50
H 2	1135	89	85	1690	87	85
I	935	94	89	1975	85	76
L1	950	76	54	1200	74	65
M1	2395	82	69	4000	72	45
N	2310	96	86	2900	99	94
Avg.	....	83	68	....	84	70
Max.	....	96	89	....	99	94
Min.	....	71	44	....	71	45

[a] Based on 100 per cent compressive strength equals strength at 70 per cent flow.
[b] Different masonry cement.
[c] Same masonry cements. Different sands.
[d] Different limes.
[e] Lime putty instead of hydrate.

The effect of change in initial flow on compressive strength is very definite. Compared with masonry cement mortars, initial flow is somewhat more uniform in its effect on strength of 1:1:6 cement-lime mortars. The two mortars are alike in that they both show a trend toward the 28-day strengths being less affected by variation in initial flow than are the 7-day strengths.

Flow of Mortars:

The wide range of strength in masonry cement mortars, as shown in Table II, was expected on account of the known variation in nature and properties of the cementitious materials. A range from 390 to 3050 psi. in the 7-day compressive strength of 1:1:6 mortars molders at a flow of 70 per cent appears greater than is reasonably explainable by differences in materials. The opinion was offered that much (not all) of this variation was explained by variation in flow.

The following question, accordingly, was addressed to each of the cooperating laboratories:

> State in terms of average, maximum, and minimum the flow of mortar from the mason's board as measured in your laboratory. The purpose of this question is to provide a rough check on flow table readings. Your comments and those of masons on the workability of the various mixes at the three flows will be appreciated.

Laboratories C, F, H, and I reported that "mason's consistency" is in the range of 105 to 120 per cent flow. Laboratory D considered that the mason would have to bring the 1:1:6 mortar to a flow of at least 135 per cent before it was workable. Correspondence with laboratory A indicates that "mason's consistency" was above 135 per cent. Laboratories G and N reported lack of workability of the low flow mortars. Laboratory M reported that the average flow of mortars made from 20 different masonry cements using sand M1 was between 100 and 105 per cent with a maximum of 125 and a minimum of 80 per cent when taken from the mason's board. There is some evidence that laboratory B had a flow table which read unusually low.

In summary, the laboratories which reported high flows for "mason's consistency" averaged much higher strength than those reporting low flows. This is consistent with the observed effects of flow on strength. Further evidence is provided by the range in amount of mixing water reported in Table III. Means for calibrating flow tables should be provided. There is evidence that one laboratory's flow of 105 per cent is another laboratory's 135 per cent and yet another's 70 per cent.

TABLE III.—COMPARISON OF AMOUNTS OF WATER ADDED TO BATCHES OF MORTAR MADE FROM 314 G. PORTLAND CEMENT, 133 G. HYDRATED LIME, AND 1500 G. SAND (1:1:6 BY VOLUME) IN DIFFERENT LABORATORIES.

Laboratory	70 per cent Flow			105 per cent Flow			135 per cent Flow		
	Water, g.	Compressive Strength, psi.		Water g.	Compressive Strength, psi.		Water g.,	Compressive Strength, psi.	
		7 days	28 days		7 days	28 days		7 days	28 days
A....	260	1880	2480	285	1520	2390	310	1360	2310
B....	353	390	555	390	295	420	427	225	355
C....	360	730	1000	389	520	710	427	320	570
D....	243	3050	3850	267	2650	3500	294	2250	2875
H2...	335	1135	1690	375	1010	1475	404	970	1440
I....	353	935	1975	378	835	1675	411	740	1500

Flow After Suction:

Not all of the laboratories reported flow after suction. Table IV summarizes the principal data from the survey. All of the masonry cement mortars tested met the proposed requirement of not less than 65 per cent of initial flow (100 to 115 per cent). The nature of the lime appears to control flow after suction. Five out of 11 hydrates pass and 6 fail on the 65 per cent requirement. The one putty mortar which was tested passed. Evidence that nature of the lime is more important than its quantity in affecting flow after suction is provided by the following data from laboratory H (these are the same mortars whose strengths were reported in the section on strength of cement-lime mortar):

TABLE IV.—FLOW AFTER SUCTION OF MASONRY MORTARS, PER CENT OF INITIAL FLOW.

Laboratory	Masonry Cement Mortars (1:3)			Cement-Lime-Sand Mortars (1:1:6)		
	70 per cent Flow	105 per cent Flow	135 per cent Flow	70 per cent Flow	105 per cent Flow	135 per cent Flow
A............	58	72	71	42	48	54
B............	..	..	..	..	..	..
C............	87	75	61	92	82	68
D............	86	95	89	83	75	89
E 1..........	..	..	..	..	..	..
E 2..........	..	..	..	..	..	..
F............	48	68	72	45	55	60
G 1[a].......	..	..	..	36	43	45
G 2[a].......	..	..	..	23	31	37
H 1[b].......	..	..	..	85	85	89
H 2..........	..	..	..	30	34	36
I............	..	..	..	..	..	..
J 1[c].......	65	76	70	..	..	..
J 2[c].......	60	70	67	..	..	..
K 1[d].......	60	73	69	..	..	..
K 2[d].......	67	62	67	..	..	..
L............	71	81	72	40	44	55
M 1[d].......	..	76	..	..	81	..
M 2[d].......	..	81	..	..	72	..
N............	83	79	70	..	65	58
Avg..........	68	76	71	53	60	59

[a] Different limes.
[b] Lime putty instead of hydrate.
[c] Different masonry cements.
[d] Same cements, different sands.

Proportions by Volume	Flow After Suction, per cent of initial flow	
	With Lime Putty	With Hydrate
1:2:9...............	82	43
1:1:6...............	81	43
1:0.2:3.............	64	37

The three mortars of 1:1:6 proportions using lime putty reported by laboratory L gave water retentions of 80, 87, and 91 per cent, respectively. Since it was stated that initial flows were not measured, it must be assumed that these values represent "absolute flows."

The foregoing data are presented as information. Comment and discussion are desired.

Respectfully submitted on behalf of the committee,

J. W. McBurney.

REPORT OF COMMITTEE C-15
ON
MANUFACTURED MASONRY UNITS

Committee C-15 on Manufactured Masonry Units held two meetings during the year: in Chicago, Ill., on June 26, 1941, during the annual meeting of the Society, and in Cleveland, Ohio, on March 3, 1942.

Since the 1941 annual meeting, two new members have been elected and one resignation accepted. The committee membership now totals 60, of whom 26 are classified as producers, 12 as consumers, and 22 as general interest members.

Subsequent to the 1941 annual meeting, Committee C-15 presented to the Society through Committee E-10 on Standards an extensive revision of the Tentative Specifications for Building Brick (Made from Clay or Shale) (C 62 – 40 T). The recommendation was accepted[1] by Committee E-10 on August 25, 1941, and the revised specifications now appear in the 1941 Supplement, Part II, under the designation C 62 – 41 T.

During the year Committee C-15 recommended that all the standard specifications and methods under its jurisdiction be submitted to the American Standards Association for approval as American standard which approval has since been granted. The ASA numbers assigned to these standards are indicated below:

Standard Specifications for:

Structural Clay Load-Bearing Wall Tile (A.S.T.M. C 34 – 41; ASA A74.1–1942),
Concrete Building Brick (C 55 – 37; A75.1–1942),
Structural Clay Non-Load-Bearing Tile (C 56 – 41; A76.1–1942),
Structural Clay Floor Tile (C 57 – 39; A77.1–1942),
Sand-Lime Building Brick (C 73 – 39; A78.1–1942),
Hollow Load-Bearing Concrete Masonry Units (C 90 – 39; A79.1–1942),
Hollow Non-Load-Bearing Concrete Masonry Units (C 129 – 39; A80.1–1942),
Concrete Masonry Units for Construction of Catch Basins and Manholes (C 139 – 39; A73.1–1942),
Solid Load-Bearing Concrete Masonry Units (C 145 – 40; A81.1–1942), and

Standard Methods of:

Sampling and Testing Brick (C 67 – 41; A82.1–1942),
Sampling and Testing Structural Clay Tile (C 112 – 36; A83.1–1942), and
Sampling and Testing Concrete Masonry Units (C 140 – 39; A84.1–1942).

RECOMMENDATIONS AFFECTING STANDARDS AND TENTATIVE STANDARDS

The committee is recommending the publication as tentative of a proposed revision of one standard and the adoption as standard of two tentative standards. Explanations of the recommended actions are given under Activities of Subcommittees. Table I lists the recommendations and gives the results of the submittal of the items to letter ballot of the committee.

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[1] In submitting this recommendation to Committee E-10 on Standards, Committee C-15 reported results of the letter ballot vote as follows: Of a total membership of 59, 46 members returned their ballots, of whom 39 voted affirmatively, 2 negatively, and 5 members marked their ballots "not voting."

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots marked "Not Voting"
I. TENTATIVE REVISION OF STANDARD			
Methods of Sampling and Testing Brick (C 67 - 41; ASA A 82.1 - 1942)	31	0	12
II. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Spec. for Sewer Brick (Made from Clay or Shale) (C 32 - 39 T)	30	0	13
Spec. for Paving Brick (C 7 - 40 T)	35	0	7

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Clay Building Brick (J. W. McBurney, chairman).—A proposed Method of Test for Initial Rate of Absorption (Suction) of Brick[2] prepared by this subcommittee as an additional test procedure for inclusion in the Standard Methods of Sampling and Testing Brick (C 67 - 41)[3] is being recommended by Committee C-15 for publication as a tentative revision of this standard. Although the standards for brick do not include requirements for rate of absorption, this property sometimes is referred to in specifications for masonry walls. For this reason it seemed desirable to have available a standard method of testing.

Subcommittee V on Paving Brick (W. F. Kellerman, chairman).—The Specifications for Paving Brick (C 7 - 40 T)[4] prepared by this subcommittee have been published during the past two years as tentative and no unfavorable comments have been received concerning them. A recommendation for the adoption as standard of these specifications has been submitted to letter ballot of the committee, subject to confirmation by the committee at its meeting during the 1942 annual meeting.

Subcommittee VI on Sewer Brick (T. R. Lawson, chairman). — Substantial agreement has now been attained as to the adequacy of the Tentative Specifications for Sewer Brick (Made from Clay or Shale) (C 32 - 39 T)[5] and they are accordingly being recommended for adoption as standard.

Subcommittee VII on Glazed Brick and Tile (J. R. Kauffman, chairman).—During the past two years considerable progress has been made by the industry on the standardization of sizes and limits on dimensional tolerances, and representatives of the industry have cooperated with the subcommittee by supplying the information needed in the preparation of revisions of the specifications for glazed building units. An extensive proposed revision of the current Tentative Specifications for Glazed Building Units (C 126 - 39 T) has been considered by the subcommittee and it is expected that this will be recommended for action during the current year.

Subcommittee VIII on Structural Clay Tile (H. C. Plummer, chairman).—The Standard Methods of Sampling and Testing Structural Clay Tile (C 112 - 36) and the Standard Definitions of Terms Relating to Structural Clay Tile (C 43 - 36) have been published without revision for six years. Inasmuch as no information is available indicating a need for revision, it is recommended that these standards be continued without change.

Working Committee on Tests for Brick Prisms (H. C. Plummer, chairman).—Tests sponsored by the committee were reported by W. J. Krefeld[6] in 1938. As no further work on the subject is contemplated at present, this Working Committee has been discharged.

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[2] This method was accepted as tentative by the Society and appears in the 1942 Book of A.S.T.M. Standards, Part II, p. 1098.

[3] 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 18.

[4] 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 183.

[5] 1939 Book of A.S.T.M. Standards, Part II, p. 925.

Working Committee on Freezing-and-Thawing Tests for Masonry Units (J. W. Whittemore, chairman).—The results of an investigation of the relations between properties of clay bricks defined in the Tentative Specifications for Building Brick (C 62 – 41 T) and performance in freezing-and-thawing tests are reported in the paper on "Relations Between Results of Laboratory Freezing and Thawing and General Physical Properties of Certain Soft-Mud Bricks," by J. W. McBurney which appears as an Appendix[7] to this report.

The present officers of the committee were re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 60 members; 41 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

D. E. PARSONS,
Chairman.

J. W. WHITTEMORE,
Secretary.

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[6] W. J. Krefeld, "Effect of Shape of Specimen on the Apparent Compressive Strength of Brick Masonry," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part I, p. 363 (1938).

[7] See p. 837.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee C-15 presented to the Society through Committee E-10 on Standards the following recommendations:

Tentative Revisions of Standard:

Methods of Sampling and Testing Brick (C 67 – 41), comprising a Method of Test for Efflorescence of Brick and

Specifications for Hollow Load-Bearing Concrete Masonry Units (C 90 – 39).

These recommendations were accepted by Committee E-10 on August 24, 1942. The tentative revision of Standard C 67, was incorporated in the new Tentative Method of Test for Initial Rate of Absorption (Suction) and Efflorescence of Brick (C 67 – 42 T) which appears in the 1942 Book of A.S.T.M. Standards, Part II, p. 1098; the tentative revision of Standard C 90 appears in the same book on p. 1440.

REPORT OF COMMITTEE C-16

ON

THERMAL INSULATING MATERIALS

Committee C-16 on Thermal Insulating Materials and its subcommittees held meetings on two occasions during the past year. One of these was on October 16, 17, and 18, 1941, at Nittany Lion Inn, State College, Pa., and the other on March 5 and 6, 1942, at the Hotel Cleveland, Cleveland, Ohio, during the spring group meetings of the Society. At the latter meeting the subcommittee arrangement that had been in force since the organization of the committee was suspended and a temporary reorganization effected in order to expedite the preparation of war-emergency specifications. In making this change an effort was made to subdivide the work of the committee among a larger number of subgroups. These temporary emergency subgroups were assigned portions of the work as follows:

On Block Insulation,
On Structural Insulating Board,
On Pipe Covering,
On Insulating Cement,
On Loose-Fill Insulation, and
On Blanket and Semi-Rigid Types of Insulation.

Subcommittee V on Thermal Properties Other Than Thermal Conductivity and Subcommittee VI on Thermal Conductivity were not included in the reorganization.

It is the intention, as soon as the National Emergency has passed, to reinstate the previous subcommittee structure in so far as this seems desirable.

At the March meeting of the committee the following officers were elected for the ensuing term of two years:

Honorary Chairman, J. H. Walker.
Chairman, E. T. Cope.
Vice-Chairman, H. H. Rinehart.
Secretary, Ray Thomas.
Advisory Committee, Walter Sinclair, H. W. Greider, and B. A. Hollenbeck.

Since the last report, the committee has gained eight new members and has lost one who has retired because of pressure of war work.

NEW TENTATIVE STANDARD

The committee recommends that the proposed Tentative Method of Test for Thermal Conductivity of Materials by Means of the Guarded Hot Plate be accepted for publication as tentative as appended hereto.[1]

This method was developed by the Joint Committee on Thermal Conductivity of all Forms of Insulation sponsored by the American Society of Heating and Ventilating Engineers, the American Society of Refrigerating Engineers, the National Research Council, and A.S.T.M. The A.S.T.M. representation on the Joint Committee is held through Subcommittee VI of Committee C-16.

Before submitting this new proposed tentative method of test to letter ballot of the membership of Committee C-16, a series of tests was conducted in each of six laboratories available to different members of the Joint Committee. Each of these laboratories was equipped with a guarded hot plate that conformed to the specifications agreed on by the joint committee and was operated as specified in the proposed method of test. Four types of material were tested and the same specimen of each was tested in two or more laboratories.

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[1] This method was accepted as tentative by the Society and appears in the 1942 Book of A.S.T.M. Standards, Part II, p. 1131.

The results obtained in all cases were in satisfactory agreement. The studies carried on in connection with this method are discussed in a report prepared by F. C. Houghten on "Conductivity Determination by the Guarded Hot-Plate Method," which appears in an Appendix to this report.

This recommendation has been submitted to letter ballot of the committee which consists of 45 members; 37 members returned their ballots, of whom 37 have voted affirmatively, 0 negatively, and 0 members marked their ballots "not voting."

ACTIVITIES OF SUBCOMMITTEES

As noted earlier in this report, the activities of Subcommittee II on Physical Properties of Preformed Block Insulation, Subcommittee III on Physical Properties of Insulating Cements, and Subcommittee IV on Physical Properties of Blanket, Flexible, Loose-Fill, and Miscellaneous Types of Insulation, active prior to the Cleveland meeting, have been suspended for the duration or until such time as reinstatement is deemed advisable. This suspends much of the study and voluntary research work that was being expended on methods of testing. The work being done was of such excellent quality that it should be continued as soon as possible.

The redistribution of the work of these three subcommittees was as follows:

Subgroup A on Physical Properties of Block Insulation (R. H. Jebens, acting chairman).—This group is collecting data on test methods from all available sources, the best of these will be adopted and temporary war emergency purchasing specifications will be prepared for presentation to the committee at its next meeting, scheduled to be held during the annual meeting of the Society in June.

Subgroup B on Structural Insulating Board (E. C. Lloyd, chairman)

Subgroup C on Pipe Covering (Ray Thomas, chairman)

Subgroup D on Insulating Cement (H. H. Rinehart, chairman)

Subgroup E on Loose Fill Insulation

Subgroup F on Blanket and Semi-Rigid Types of Insulation (R. E. Cryor, chairman)

All the subgroups pursued the same plan as that outlined for Subgroup A and all are expecting to offer Emergency Specifications at the next meeting of the committee.

Subcommittee VI, the A.S.T.M. Group of the Joint Committee on Thermal Conductivity of all Forms of Insulation (F. C. Houghten, chairman).—This subcommittee held two meetings during the past year. The Joint Committee is sponsored by the A.S.H.V.E., A.S.R.E., N.R.C., and A.S.T.M. The program of testing to determine whether the values of thermal conductivity of given specimens as obtained by different cooperating laboratories using the Proposed Tentative Method of Test for Thermal Conductivity of Materials by Means of the Guarded Hot Plate, would be in satisfactory agreement was reported on and the results submitted to the committee. These results showed that the proposed method contained the essential elements necessary for obtaining satisfactory results by its use.

This subcommittee further started a study of the guarded hot box method for the determination of thermal conductivity of walls and other fabricated heat barriers.

Subcommittee VII on Dimensional Standards for Preformed Insulation (Ray Thomas, chairman).—This subcommittee has collected and reported on a great mass of data and has proposed elimination of certain present dimensions now in use. These proposals have been submitted to the manufacturers for comment and this whole matter of dimensional standards may ultimately be handled solely within the industry.

Subcommittee on Editing (C. B. Bradley, chairman).—As mentioned in last year's report, this subcommittee rarely holds formal meetings but does its work by correspondence, suggesting changes in form and style of proposed methods so that they conform to A.S.T.M. standard practice.

Subcommittee for Liaison with A.S.T.M. Committee C-8 on Refractories (R. H. Heilman, chairman).—This subcommittee has held no meetings during the past year as no matters requiring adjustment between Committees C-8 and C-16 have arisen.

This report has been submitted to letter ballot of the committee which consists of 45 members; 37 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. H. WALKER,
Chairman.

E. T. COPE,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee C-16 presented to the Society through Committee E-10 on Standards the following recommendations under the emergency procedure:

Emergency Specifications for:

- 85 per cent Magnesia Thermal Insulating Cement (ES-8),
- Long-Fiber Asbestos Thermal Insulating Cement (ES-9),
- Mineral Wool Thermal Insulating Cement (ES-10),
- Expanded or Exfoliated Mica Thermal Insulating Cement (ES-11),
- Diatomaceous Silica Thermal Insulating Cement, for Use from 600 to 1200 F. (ES-12),
- Diatomaceous Silica Thermal Insulating Cement, for Use from 1200 to 1900 F. (ES-13),
- Blanket Thermal Insulation for Building Purposes (ES-14),
- Blanket Thermal Insulation for Industrial Purposes (ES-15),
- Blanket Thermal Insulation for Refrigeration Purposes (ES-16),
- Preformed Pipe Covering Thermal Insulation (ES-17),
- Preformed Block Thermal Insulation (ES-18), and
- Structural Board Insulation (Thermal Insulation) (ES-19).

These new emergency specifications were accepted by Committee E-10 on August 24, 1942, and assigned the A.S.T.M. serial designations indicated above. They appear in the 1942 Book of A.S.T.M. Standards, Part II, pp. 1026 to 1050.

APPENDIX

CONDUCTIVITY DETERMINATION BY THE GUARDED HOT-PLATE METHOD

A report on behalf of the Joint Committee on Thermal Conductivity of the A.S.H.V.E., A.S.T.M., A.S.R.E., and the National Research Council[1]

PREPARED BY F. C. HOUGHTEN[2]

In the codification of any testing method there is always the question as to whether the method and apparatus to be used should be limited to a single set procedure or design, or whether the procedure to be followed and the specifications of the apparatus to be used should be limited only in so far as is necessary to obtain the desired result, leaving to the individual laboratory as much discretion in design and procedure as possible. In the development of the code[3] by the Joint Committee on Thermal Conductivity[1] this question was given very careful consideration, with the result that the committee agreed on the latter course, namely, that the standard method should outline principles in procedure and necessary limitation in the design of the apparatus to be used, and leave to the discretion of the engineer making the test as much latitude as possible.

The reasons for the wide latitude permitted were more compelling than is usually the case since the test is designed for use on materials having conductivities ranging up to 5.0 Btu. per sq. ft. per hr. per deg. Fahr. per in. and over the extremely wide temperature range from −50 to 1400 F. Further, the test procedure is designed for the testing of rigid board material, for flexible materials including blankets, and for fill materials. While a single test procedure, including the design of the apparatus to be used, might conceivably be developed, a wide latitude is highly desirable. As an example, for the determination of conductivities in the low and moderate temperature fields many materials are available for the construction of the plate itself, since deterioration from heating to high temperature will not occur, while for testing of materials in the upper part of the −50 to 1400 F. range few materials are available. Again, a guarded hot plate that is to be used only for the determination of conductivity of insulating materials having low conductivities, say between 0.25 and 0.50, will require a low energy transformation rate in the heater, while if the plate is to be used for testing materials having higher conductivity the energy transformation rate must be greater and the wiring of the heater coils different. Further, in order to permit experimentation in the operation of the test, it seemed desirable to allow the individual as much latitude as possible.

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[1] The personnel of the Joint Committee consists of the following: F. C. Houghten, Chairman, C. B. Bradley, H. C. Dickinson, H. W. Eagles, R. H. Heilman, E. R. Queer, F. B. Rowley, T. S. Taylor, and G. B. Wilkes.

The A.S.T.M. representatives on the Joint Committee comprise Subcommittee VI of Committee C-16 on Thermal Insulating Materials, together with Messrs. J. D. Babbitt, Ralph Bowman, L. K. Jones and H. G. Knode.

[2] Director of Research, American Society of Heating and Ventilating Engineers, Pittsburgh, Pa.

[3] The Code for Testing Thermal Conductivity by Means of the Guarded Hot Plate has been accepted by the Joint Committee and is now being considered for adoption by the A.S.T.M. and A.S.H.V.E.

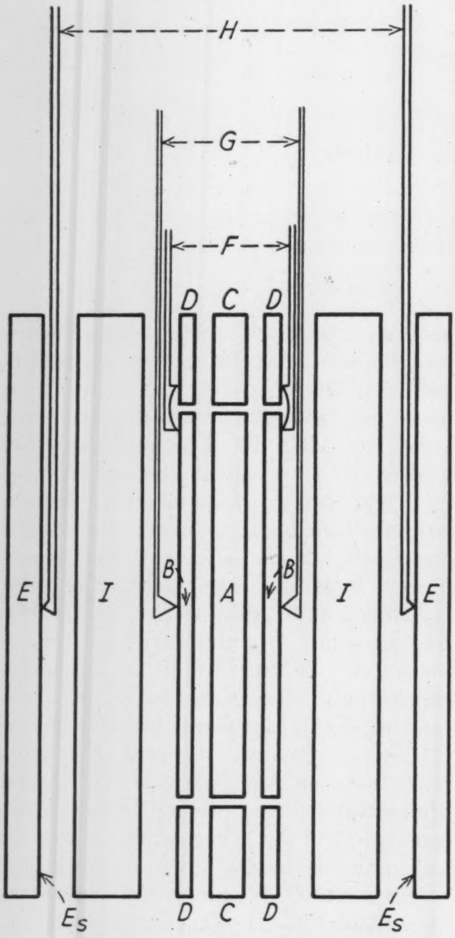

FIG. 1.—Schematic Diagram Showing General Features of the Guarded Hot Plate.

A = Central Heater.
B = Central surface plates. *A* and *B* comprise the central section of heating unit.
C = Guard heater.
D = Guard surface plates. *C* and *D* comprise the guard section of heating unit. *A*, *B*, *C*, and *D* comprise the heating unit.
E = Cooling units.
E_s = Cooling unit surface plates.
F = Differential thermocouples.
G = Heating unit surface thermocouples.
H = Cooling unit surface thermocouples.
I = Test specimen.

To those particularly interested in the comparability of the test results, it may seem that this wide latitude might defeat the real purpose of the code; that is, it may appear that two laboratories operating under the limitations of the code and using variations in the apparatus and performance permitted might be expected to obtain different results for a given material. While the members of the committee had complete confidence that the limitations imposed would permit no such variations, it nevertheless agreed that a series of tests (on the same samples of a number of types of materials) should be made by different laboratories, all operating under the code. This paper therefore deals with this program and the results obtained.

THE GUARDED HOT PLATE

The test procedure does not include detailed specifications for the construction or operation of any particular guarded hot plate for determining conductivity values. Any plate complying with the limitations set forth will be satisfactory. For the convenience of new workers in the field, arrangements have been made for making available complete specifications and directions for the building and use of three typical guarded hot plates complying with the requirements of the code and known as the National Bureau of Standards plate, the National Research Council plate, and the Alundum plate. The National Bureau of Standards plate and the National Research Council plate are square and the heated surfaces are of metal. The Alundum plate is built with the heating coils molded into alundum cement so as to give a circular plate with alundum cement surfaces. It has the obvious advantage of being usable in the moderately high-temperature field.

The general features of the guarded hot plate are shown in Fig. 1. Plates are usually square, but round plates are occasionally used. The term "guarded hot plate" is applied to the entire assembled apparatus, including the heating unit and the cooling units. The heating unit consists of two sections: a central section and a guard section. The central section consists of a central heater and two central surface plates. The surface plates are usually made of noncorrosive, highly conducting metal; however, the central section and the guard section of the heating unit may each be molded of alundum cement or other materials of low conductivity so as to include both the heater and the surface plates. The guard section consists of one or more guard heaters and the guard surface plates, except that both the heater and the surface plates may be one piece when alundum cement or other material of low thermal conductivity is used.

In the hot plate shown in Fig. 1, heat is supplied electrically to the heating sections, *A* and *C*. Since *C* surrounds and "guards" against radial heat flow from *A*, that generated within *A* is permitted to flow only laterally through *B*—*B* and thence through the two identical samples of material between *E* and *B*. The requirements for a satisfactory test are met when this condition is realized within the limits of accuracy required: namely, when all of the heat generated in *A* flows uniformly and laterally in both directions in straight and parallel lines through *B*, thence through the two samples of insulating materials to the heat absorber *E*.

The committee recognized the need of basing certain limitations in design upon the character of the material to be tested and its unit conductance. As an example, for nonrigid materials, whose surface would conform to the surfaces of *B* and *E*, and which have unit conductances not to exceed 1.0 Btu. per sq. ft. per hr. per deg. Fahr. per in., the temperature of the surfaces of *B* and *E* may be assumed to be at the temperature of the material under test; while for rigid materials not conforming readily to the surfaces of *B* and *E*, and for all materials having a unit conductance exceeding 1.0 Btu. per sq. ft. per hr. per deg. Fahr. per in., the surface temperatures must be determined by thermocouples having their junctions immediately in contact with the test sample.

VARIATIONS IN TEST SAMPLES

Members of the Joint Committee were further confident that the variations in conductivity values obtained by different laboratories under past practices resulted more from the selection and treatment of the sample under test than for any other reason. The code therefore goes to considerable length in the selection of the samples, in fixing the number of samples of a given material to be tested, and the number of individual tests to be made on each of these samples, in order to insure that the results obtained from the test reasonably represent a given material. The code

TABLE I.—INSULATION MATERIALS TESTED AT DIFFERENT LABORATORIES BY THE USE OF DIFFERENT TYPES OF PLATE.

Material	Laboratory	Type of Plate
Low-conductivity inorganic fill	B	Bureau of Standards, 18 in square
	A	Alundum, 8⅛ in. round
	C	National Research Council, 12 in. square
Low-conductivity inorganic board	B	Bureau of Standards, 18 in square
	E	National Research Council, 12 in. square
	D	Alundum, 9⅜ in. round
Low-conductivity organic board	C	National Research Council, 12 in. square
	E	National Research Council, 12 in. square
	F	Bureau of Standards, 18 in. square
High-conductivity inorganic board	B	Bureau of Standards, 18 in. square
	E	National Research Council, 12 in. square
	D	Alundum, 9⅜ in. round

TABLE II.—DATA RELATING TO TESTS ON LOW CONDUCTIVITY INORGANIC FILL INSULATION.

Mean Temperature, deg. Fahr	Value of Conductivity K Obtained from Test	Test Value of K, per cent of Value Obtained from Mean Curve Fig. 2	Departure of Test Value of K from Mean Curve, per cent	Laboratory Making Test
79.0	0.540	100.0	0	B
79.6	0.541	100.2	+0.2	C
79.75	0.547	101.3	+1.3	C
104.5	0.565	101.3	+1.3	B
109.0	0.561	100.0	0	C
109.2	0.553	98.6	−1.4	C
132.8	0.580	100.7	+0.7	B
137.1	0.568	98.3	−1.7	C
138.5	0.563	97.4	−2.6	C
157.6	0.605	102.4	+2.4	B
228.0	0.617	97.9	−2.1	A
241.0	0.659	103.6	+3.6	B
320.0	0.650	98.8	−1.2	A

further specifies at considerable length the treatment of the sample and the placing of it in the apparatus, including the condition of dryness, the temperature at which it had been previously heated, the weighing, and measuring of its thickness.

TEST PROGRAM AND RESULTS

The program of tests included the use of each of the three plates mentioned above. Six laboratories participated in the program, using two Bureau of Standards plates, 18 in. square, two National Research Council plates, 12 in. square, and two Alundum round plates, $8\frac{1}{8}$ in. and $9\frac{3}{8}$ in. in diameter, respectively. The materials tested covered a wide range in conductivity and maximum temperature to which they might be used. The materials tested and the type and size of plates used are given in Table I.

In the case of each insulating material tested, samples were chosen and heat treated to or above the maximum temperature at which they might be tested. These samples were then carefully guarded in shipment from one laboratory to another, and each laboratory tested the sample following all the provisions of the test code, including the drying of the sample. In each case the material was first tested by the laboratory requiring the largest area or lateral dimensions of the sample; the next laboratory testing the sample cut it down to the size required by its apparatus. For the single fill material tested, a sufficient amount of the sample was chosen, heat treated, and thoroughly mixed; this

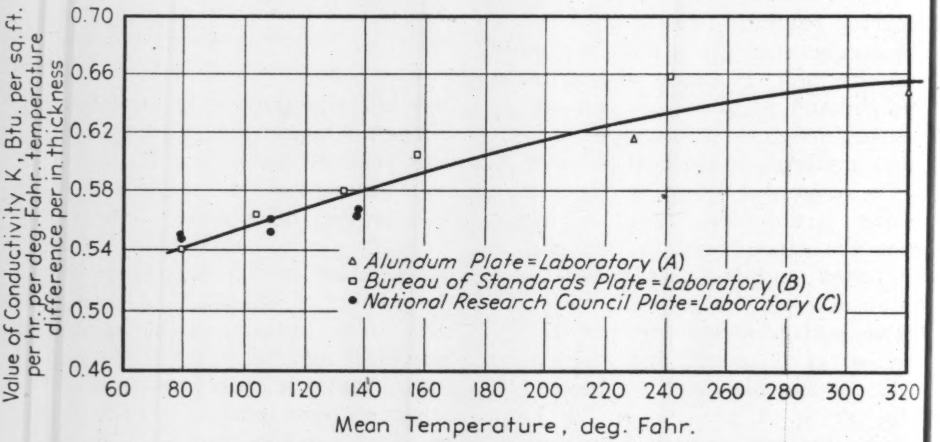

FIG. 2.—Conductivity-Mean Temperature Curves for Low-Conductivity Inorganic Loose-Fill Insulation.

TABLE III.—DATA RELATING TO TESTS ON LOW-CONDUCTIVITY INORGANIC BOARD INSULATION.

Mean Temperature, deg. Fahr.	Value of Conductivity K Obtained from Test	Test Value of K, per cent of Value Obtained from Mean Curve Fig. 3	Departure of Test Value of K from Mean Curve, per cent	Laboratory Making Test
31.6......	0.484	97.6	−2.4	E
70.0......	0.507	97.9	−2.1	E
79.0......	0.532	101.9	+1.9	B
122.4......	0.555	102.0	+2.0	B
142.2......	0.537	97.1	−2.9	E
163.0......	0.572	101.6	+1.6	D
175.5......	0.576	101.2	+1.2	B
181.4......	0.566	99.0	−1.0	E
246.0......	0.600	99.8	−0.2	D
294.1......	0.621	99.8	−0.2	B
309.0......	0.632	100.5	+0.5	D
314.0......	0.631	100.0	0	D
364.0......	0.654	100.3	+0.3	D
371.0......	0.657	100.5	+0.5	D
442.0......	0.679	99.6	−0.4	D
447.0......	0.682	99.7	−0.3	D
447.0......	0.677	99.0	−1.0	D
504.0......	0.709	100.6	+0.6	D
510.0......	0.711	100.6	+0.6	D
516.0......	0.706	99.6	−0.4	D
565.0......	0.726	100.0	0	D
640.0......	0.746	99.9	−0.1	D
669.0......	0.752	99.6	−0.4	D
672.0......	0.758	100.3	+0.3	D

amount was then divided into three parts for distribution among the three laboratories making the test.

All test results from each individual laboratory were kept confidential and sent to the Research Laboratory of the American Society of Heating and Ventilating Engineers, where the Chairman of the Joint Committee compared them and plotted the data as shown in Figs. 2 to 5. Each point for the different materials plotted in Figs. 2 to 5 represents a single test at a given mean temperature made by one of the participating laboratories; curves were drawn to represent the best fit for all the tests of a given material.

Tabulated data, Tables II to V, in connection with the curve for each material give a comparison of the conductivities found by each individual test and that of the common curve for the material. It will be observed that the point showing maximum deviation from the common curve value was +3.6 per cent for low-conductivity inorganic

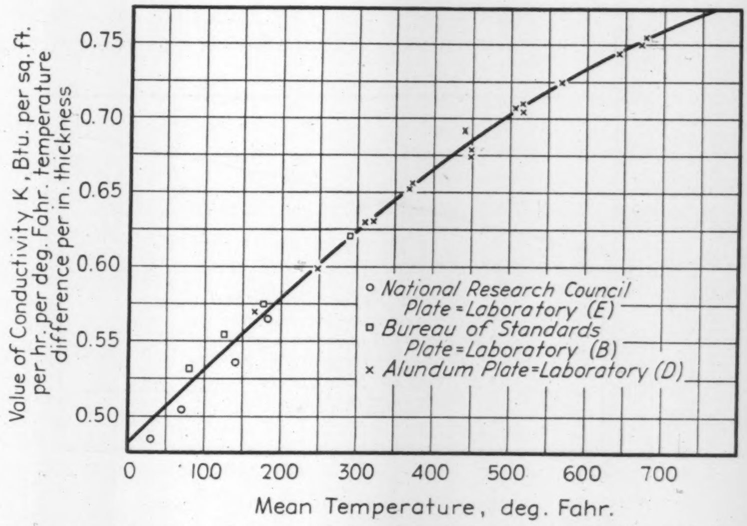

FIG. 3.—Conductivity-Mean Temperature Curves for Low-Conductivity Inorganic Insulating Board.

TABLE IV.—DATA RELATING TO TESTS ON LOW-CONDUCTIVITY ORGANIC FIBERBOARD INSULATION.

Mean Temperature, deg. Fahr.	Value of Conductivity K Obtained from Tests	Test Value of K, per cent of Value Obtained from Mean Curve, Fig. 4	Departure of Test Value of K from Mean Curve, per cent	Laboratory Making Test
31.5....	0.345	101.8	+1.8	E
70.0....	0.362	101.1	+1.1	E
71.0....	0.357	99.7	−0.3	F
75.1....	0.353	98.1	−1.9	F
80.0....	0.362	100.0	0	F
83.6....	0.360	98.9	−1.1	F
84.0....	0.364	100.0	0	F
93.2....	0.365	98.9	−1.1	F
93.4....	0.370	100.3	+0.3	F
93.7....	0.366	99.2	−0.8	F
100.5....	0.368	98.9	−1.1	C
101.0....	0.365	98.1	−1.9	C
124.0....	0.384	100.3	+0.3	E
184.9....	0.411	99.8	−0.2	E

fill, −2.9 per cent for low-conductivity inorganic board, −1.9 per cent for low-conductivity organic board, and +6.6 per cent for high-conductivity inorganic board, while the average deviations of all the individual test points for the four representative materials were 1.42, 0.87, 0.85, and 4.2 per cent, respectively.

Except for the high-conductivity material, which is recognized as being more difficult to test, the check was unusually good, while even for the high-conductivity material, the deviation from the average for the worst test was only +6.7 per cent, while the average deviation was +4.2 per cent; thus adequately justifying the point of view of the committee in permitting as much variation as was considered to be consistent with the requirements of the code, and indicating clearly that where the test procedure laid down in the code was followed the resulting average curve drawn gives values accurate to a very small percentage.

The test results plotted in Figs. 2 to 5 further indicate certain desirable requirements in the satisfactory testing of a given material in order to make it possible to draw curves showing the conductivity of the material over a wide mean temperature range. The four test points at mean temperatures of 80, 90, 92, and 100 F. by Laboratory B for high-conductivity inorganic structural insulating board indicate the desirability of spreading such tests over a wider mean temperature range covering the entire range from which conductivity is to be predicted; that is, the small percentage variation in the conductivity indicated by different tests when made over a narrow range in mean

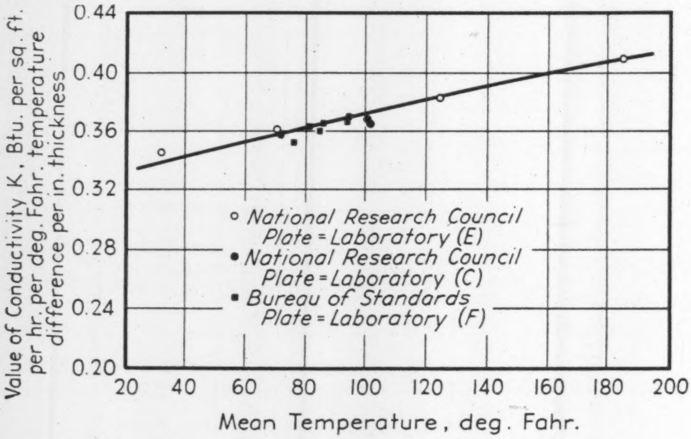

FIG. 4.—Conductivity-Mean Temperature Curves for Low-Conductivity Organic Fiber Insulating Board.

temperature may so fall as to indicate a conductivity-mean temperature curve that, if extrapolated, would give highly erroneous results.

The test results also show the fact assumed by the committee, that the variations obtained by tests either by the same laboratory or by different laboratories for a given material, will depend more upon choice and treatment of the sample than upon the procedure of the test and the apparatus used. It is probable that the differences between carefully selected samples of each of the four materials would be as great as variation in individual values obtained for the same sample tested in the different laboratories; or, in other words, the results show that the code proposed is probably more accurate in giving comparable results than the manufacturer's control in manufacturing.

TABLE V.—DATA RELATING TO TESTS ON HIGH-CONDUCTIVITY INORGANIC STRUCTURAL BOARD.

Mean Temperature, deg. Fahr.	Value of Conductivity *K*, Obtained from Tests	Test Value of *K*, per cent of Value Obtained from Mean Curve, Fig. 4.	Departure of Test Value of *K* from Mean Curve, per cent	Laboratory Making Test
23.5.....	3.75	104.5	+4.5	E
59.6.....	3.90	106.0	+6.0	E
61.2.....	3.85	104.3	+4.3	E
65.0.....	3.50	94.6	−5.4	D
67.0.....	3.50	94.3	−5.7	D
79.5.....	3.52	94.1	−5.9	B
82.5.....	3.55	94.7	−5.3	D
84.0.....	3.59	95.7	−4.3	D
89.0.....	3.59	95.5	−4.5	D
90.2.....	3.69	97.9	−2.1	B
92.5.....	3.71	98.4	−1.6	B
94.0.....	3.61	95.5	−4.5	D
100.5.....	3.79	100.0	0	B
115.0.....	3.66	95.6	−4.4	D
117.0.....	3.63	94.5	−5.5	D
131.7.....	4.01	103.4	+3.4	E
134.6.....	3.92	100.8	+0.8	E
134.7.....	4.14	106.4	+6.4	E
140.0.....	3.69	94.6	−5.4	D
141.0.....	3.71	95.1	−4.9	D
142.0.....	3.75	95.9	−4.1	D
171.1.....	4.22	106.0	+6.0	E
171.1.....	4.23	106.3	+6.3	E
172.5.....	3.95	99.0	−1.0	E
208.4.....	4.23	103.7	+3.7	E
208.5.....	4.19	102.7	+2.7	E
209.4.....	4.35	106.6	+6.6	E
209.4.....	3.98	97.5	−2.5	E

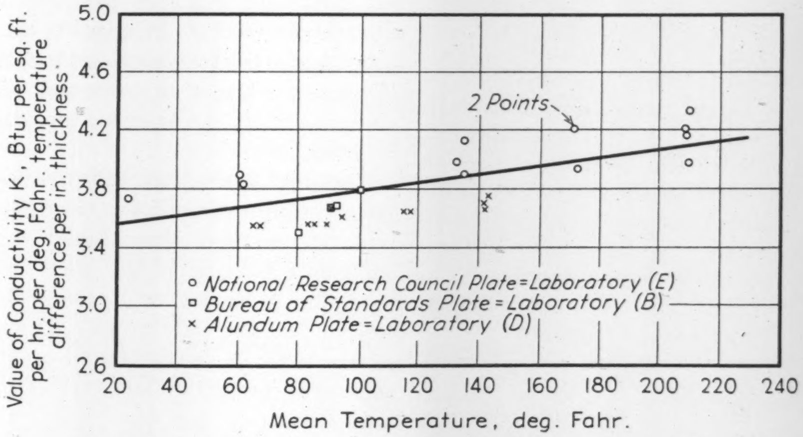

FIG. 5.—Conductivity-Mean Temperature Curves for High-Conductivity Inorganic Structural Board.

DISCUSSION

Mr. E. T. Cope[1] (*presented in written form*).—Satisfactory agreement between the values obtained by several investigators when testing a given specimen of a material is the strongest evidence obtainable to confirm the correctness of the procedure followed. This applies to Mr. Houghten's paper. The fact that it was not found necessary to make any change in the Proposed Tentative Method of Test for Thermal Conductivity by Means of the Guarded Hot Plate as a result of the tests reported on in the paper confirms the wisdom of the choice of approach emphasized by Mr. Houghten. Some of those interested in the preparation of this test method felt that a specific design of hot plate was the best solution of the problem. Had the field been virgin, this would have been the logical procedure. However, there were already several experimenters in this country who were obtaining results that were in good agreement but whose guarded hot plates differed somewhat in design. Obviously some of these devices would not have conformed to a specific design. The tests show that a specific design was not warranted.

The Joint Committee on Thermal Conductivity of All Forms of Insulation, by pursuing the course it has followed, placing the emphasis on preparation of specimens and on the operation of the test, has avoided the pitfalls of controversy and has produced a test procedure that has been shown by the tests to yield results that are in satisfactory agreement.

The consumer group is greatly interested in this test procedure. In the minds of some insulation men among this group, thermal conductivity is the most important characteristic of heat insulation. While the writer cannot go all the way with these engineers in this particular, still when he uses values of thermal conductivity he wants to be sure what degree of confidence can be placed in them. This is the demand of all consumer engineers when heat insulation is considered.

The joint committee has produced a method of test embodied in the proposed tentative method that should inspire confidence because it has gone further than the preparing of a code—it has proved that the code works. The committee is to be congratulated upon the successful completion and verification of this task.

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[1] Chairman, Committee C-16 on Thermal Insulating Materials; Research Dept., The Detroit Edison Co., Detroit, Mich.

REPORT OF COMMITTEE C-18
ON
NATURAL BUILDING STONES

Due to the fact that some members of this committee have been devoting practically all of their time to emergency work, no meetings of Committee C-18 on Natural Building Stones were held during the past year. There was considerable activity in revising definitions for various types of stone, formulating test procedures, and preparing specifications for surface finishes.

There have been two resignations in the producer group. The committee now consists of 21 members, classified as follows: 8 producers, 7 consumers, and 6 general interest members.

Three members of this committee were asked to attend a conference at the National Museum on December 19, 1941, the purpose of which was to offer suggestions in regard to a suitable disposition of the building stone collection. Excessive loads on the floors and demand for more space made it necessary for the Museum to discard some of the heavy displays. A suggestion by some of the members of this committee, that the stone collection be turned over to the National Bureau of Standards for research purposes, was accepted, and the collection has been moved to storage until conditions will permit the construction of an exposure-test wall. Since the collection contains representative samples from practically all of the important building stone deposits of this country and foreign countries, it is expected that the test will supply considerable information on the durability of various stones. This committee is cooperating in the plans for the test and will follow the results with great interest.

Subsequent to the annual meeting, Committee C-18 presented to the Society, through Committee E-10 on Standards, a new Proposed Method of Test for Compressive Strength of Natural Building Stone. This method was accepted[1] by Committee E-10 for publication as tentative on October 23, 1941, and appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 275, bearing the A.S.T.M. serial designation C 170 - 41 T.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Nomenclature and Definitions (L. W. Currier, chairman).—One meeting of this subcommittee was held since the last report was presented. Definitions were formulated for granite, sandstone, limestone, brownstone, marble, and dolomite. It was not found possible to formulate a restrictive definition for granite that would include all of the products now marketed as "granite," so it was decided to define separately the terms, commercial granite and black granite. A similar difficulty has been found in defining the term marble due to the fact that serpentine and travertine are commonly known in the trade as marble. The definitions of the above terms have been submitted to the members of the entire committee for comment, and a few criticisms were received with respect to the definitions of granite and marble. Further attention is being given to these terms in the hope of making the definitions generally acceptable.

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[1] In submitting this recommendation to Committee E-10 on Standards, Committee C-18 reported results of the letter ballot vote as follows: Of a total membership of 23, 19 members returned their ballots, of whom 17 voted affirmatively, 0 negatively, and 2 members marked their ballots "not voting."

Since some of the limestones possess certain characteristics of marble, it is difficult to establish a satisfactory demarcation between the two types. The commonly accepted distinction is that marble will take a polish while limestone will not. This criterion is not an entirely satisfactory means of distinguishing the two materials, because some of the limestones will take a low grade polish almost equal to that of some of the marbles. For this reason some attention has been given methods of evaluating different degrees of polish. Through the generous cooperation of R. J. Shank and Mr. Schwartz, of Ohio State University, measurements were obtained on nine polished samples of stone of various colors and degrees of polish. The apparatus was designed for measuring the smoothness of finish on metals, and the results are based on surface reflectance. Due to the translucency of many marbles, there is a variable amount of subsurface reflection, depending largely on the color, and this makes the results on marbles rather uncertain. Two other methods were tried on a few samples to get a comparsion between the sharpness of image and the specular reflectance tests. The sharpness of image method seems to afford a satisfactory means of evaluating polish on stone, but a large number of measurements on various materials would be necessary to establish suitable limits.

Subcommittee III on Test Procedures (D. W. Kessler, chairman).—The test procedure for determining compressive strength of stone was revised to meet certain objections and approved by letter ballot of Committee C-18. On October 23, 1941, this proposed Method of Test for Compressive Strength of Natural Building Stone was accepted by Committee E-10 for publication as tentative and issued under the designation C 170 - 41 T, as mentioned earlier in this report.

This subcommittee has also devoted some time to the study of hardness tests to determine a suitable procedure for a hardness test method.

Subcommittee IV on Quality Specifications (W. C. Clark, chairman).—This subcommittee has held no meeting during the past year, but so far as possible is cooperating in an investigation at the National Bureau of Standards to determine a method to distinguish slates of the fading and nonfading varieties. The outcome of this investigation will probably have a bearing on the future operations of this subcommittee with respect to specifications for roofing slate, and the results are being followed with interest.

Subcommittee V on Surface Finishes (H. L. Smith, chairman).—This subcommittee has made further studies of surface finishes on various types of stones. The granite industry has supplied specifications for nine types of finish on their product and all of these types are being studied looking toward their inclusion in an A.S.T.M. standard.

The present officers of the committee were re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 21 members; 17 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

WALTON C. CLARK,
Chairman.

D. W. KESSLER,
Secretary.

REPORT OF COMMITTEE D-1

ON

PAINT, VARNISH, LACQUER, AND RELATED PRODUCTS

Committee D-1 on Paint, Varnish, Lacquer, and Related Products held two meetings during the year: on June 25, 1941, in Chicago, Ill., during the annual meeting of the Society, and on March 6, 1942, in Cleveland, Ohio, during the spring group meetings of the Society.

Committee D-1 at the last two meetings arranged for the presentation of talks by J. B. Davis and M. Rea Paul on the activities of the War Production Board in the protective coating field, and also a talk by John Iliff on the studies being conducted by Subcommittee XXIX on Painting of Structural Iron and Steel, of Committee D-1.

During the past year two new subcommittees have been appointed, Subcommittee III on Bituminous Emulsions with E. F. Hickson as chairman, and Subcommittee IV on Traffic Paints with Anthony Skett as chairman. Subcommittee XXVIII on Specifications for Spar Varnish has been discontinued.

Clinton Grove has been appointed as the representative of Committee D-1 on Subcommittee XIII on Hydrogen Ion Determination, of Committee E-1 on Methods of Testing.

During the year, Committee D-1 has recommended submittal by the Society of four of its standards covering shellac to the American Standards Association for approval as American standard. Thirty-five standards under the jurisdiction of Committee D-1 have been approved by the American Standards Association as American standards.

Since the last annual meeting, 28 new members have been elected, 8 resignations have been accepted, and 2 deaths reported, leaving a total membership of 284.

Of the one adviser and sixteen subcommittees, two furnished reports of progress and eleven submitted detailed reports affecting standards, which have resulted in the committee making the recommendations given below to be acted upon at the annual meeting.

In this report, recommendations affecting standards and tentative standards are noted in brief form below, together with the results of the letter ballot. These recommendations are explained in the reports of the subcommittees directly responsible for them.

EMERGENCY PROVISION AND STANDARD

As mentioned later in this report, the committee submitted, under the new emergency procedure, an Emergency Alternate Provision in the Standard Specifications for Basic Sulfate White Lead (D 82 – 41) as well as an Emergency Method for Conducting Salt Spray Tests on Organic Protective Coatings. These two recommendations were approved by Committee E-10 on March 27 and April 6, 1942, respectively. Emergency Provision EA – D 82 was issued in the form of a pink sticker for attachment to the standard.[1] The emergency method under the designation ES – 3 was also reprinted in separate pamphlet form.

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[1] See Editorial Note, p. 293.

I. NEW TENTATIVE STANDARDS

The committee submits for publication as tentative the following two new specifications and three methods of test:

Specifications for:[2]

C. P. Toluidine Toner, proposed by Subcommittee XV,

Chrome Yellow and Chrome Orange[2], to replace immediately the Standard Specifications D 211 - 41 which are recommended for withdrawal, proposed by Subcommittee XV.

Methods of:[2]

Test for Abrasion Resistance of Organic Coatings with the Air Blast Abrasion Tester, proposed by Subcommitte XXV,

Test for Spectral Characteristics and Color of Objects and Materials, to replace immediately the present Standard D 307 - 39 which is recommended for withdrawal, proposed by Subcommittee XVIII, and

Test for Consistency of Exterior House Paints and Enamel-Type Paints, to replace the present Standard D 562 - 41 which is recommended for withdrawal, proposed by Subcommittee VIII.

II. REVISIONS OF TENTATIVE STANDARDS

The committee recommends that the following tentative method of test and definitions be revised as referred to in the reports of the subcommittee and continued as tentative:

Tentative Methods of:[3]

Sampling and Testing Aluminum Powder and Paste (D 480 - 41 T), proposed by Subcommittee VIII, and

Tentative Definitions of:[3]

Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16 - 41 T), proposed by Subcommittee VI.

III. REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption, revisions in the following standard, and accordingly asks for a nine-tenths affirmative vote at the annual meeting in order that these modifications may be referred to letter ballot of the Society:

Standard Methods of:[4]

Sampling and Testing Lacquer Solvents and Diluents (D 268 - 41), proposed by Subcommittee XXV.

IV. ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the following tentative standards be approved for reference to letter ballot of the Society for adoption as standard, and that the one so indicated be revised as referred to in the report of the subcommittee:

Tentative Specifications for:[5]

Barium Sulfate Pigments (D 602 - 41 T), proposed by Subcommittee XV,

Mica Pigment (D 607 - 41 T), proposed by Subcommittee XV,

Diatomaceous Silica Pigment (D 604 - 41 T), proposed by Subcommittee XV,

Aluminum Silicate Pigment (D 603 - 41 T), proposed by Subcommittee XV,

Lead Titanate (D 606 - 41 T), proposed by Subcommittee XV, and

Magnesium Silicate Pigment (D 605 - 41 T), revised as proposed by Subcommittee XV.

V. WITHDRAWAL OF STANDARDS

The committee recommends the withdrawal of the following standards for the reasons referred to in the reports of the subcommittees:

Standard Specifications for Chrome Yellow (D 211 - 41),[6] proposed by Subcommittee XV,

Standard Methods of Test for:

Spectral Apparent Reflectivity of Paints (D 307 - 39),[7] proposed by Subcommittee XXV, and

Consistency of Enamel Type Paints (D 562 - 41),[6] proposed by Subcommittee VIII.

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[2] These specifications and methods were accepted as tentative by the Society and appear in the 1942 Book of A.S.T.M. Standards, Part II, pp. 1343, 1341, 1368, 1374, 1372, respectively.

[3] 1941 Supplement to Book of A.S.T.M. Standards, Part II, pp. 378, 406.

[4] 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 222.

[5] *Ibid.*, pp. 373, 377, 374, 372, 375, 376.

[6] *Ibid.*, pp. 190, 237.

[7] 1939 Book of A.S.T.M. Standards, Part II, p. 815.

These recommendations have been submitted to letter ballot of the committee which consists of 222 voting members, 88 members returned their ballots, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Spec. for C. P. Toluidine Toner	47	0	41
Spec. for Chrome Yellow and Chrome Orange, to replace Standard D 211 - 41	53	0	35
Test for Abrasion Resistance of Organic Coatings with the Air Blast Abrasion Tester	54	0	34
Test for Spectral Characteristics and Color of Objects and Materials, to replace Standard D 307 - 39	51	0	37
Test of Consistency of Exterior House Paints and Enamel Type Paints, to replace Standard D 562 - 41	56	0	32
II. REVISIONS OF TENTATIVE STANDARDS			
Sampling and Testing Aluminum Powder and Paste (D 480 - 41 T)	52	0	36
Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16 - 41 T)	66	0	22
III. REVISION OF STANDARD, IMMEDIATE ADOPTION			
Sampling and Testing Lacquer Solvents and Diluents (D 268 - 41)	55	0	33
IV. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Spec. for Barium Sulfate Pigments (D 602 - 41 T)	56	0	32
Spec. for Mica Pigment (D 607 - 41 T)	52	0	36
Spec. for Diatomaceous Silica Pigment (D 604 - 41 T)	55	0	33
Spec. for Aluminum Silicate Pigment (D 603 - 41 T)	53	0	35
Spec. for Lead Titanate (D 606 - 41 T)	56	0	32
Spec. for Magnesium Silicate Pigment (D 605 - 41 T), as revised	54	0	34
V. WITHDRAWAL OF STANDARDS			
Spec. for Chrome Yellow (D 211 - 41; ASA K 27 - 1941)	51	0	37
Test for Spectral Apparent Reflectivity of Paints (D 307 - 39)	51	0	37
Test for Consistency of Enamel Type Paints (D 562 - 41)	59	0	29

ACTIVITIES OF SUBCOMMITTEES

Underground Pipe Protection (F. N. Speller, adviser).—The American Gas Association and the American Petroleum Institute are considering a new program for an investigation of underground pipe protection.

Subcommittee II on Drying Oils (Francis Scofield, chairman).—This subcommittee is investigating the demand for specifications covering alkali refined linseed oil for varnish making and for the so-called "synthetic" oils.

Subcommittee VI on Definitions (C. H. Rose, chairman).—This subcommittee recommends that the Tentative Definitions of Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16 - 41 T), be revised by the addition of the following definitions:

Lacquer.—A liquid composition containing cellulose esters or ethers as the basic film-forming ingredients. Lacquer dries rapidly by solvent evaporation.

Sanitary or Tin Plate Lacquers.—Usually varnish coatings for application on the interior of metal food containers.

Putty.—A stiff, dough-like material consisting of pigment and vehicle, used for setting window panes and filling imperfections in wood or metal surfaces.

Bituminous Varnish.—A dark colored varnish containing bituminous ingredients. The varnish may be either of the oil or spirit type.

Glaze.—A very thin coating of a paint product usually a semi-transparent coating tinted with Van Dyke brown, burnt sienna or a similar pigment; applied on a previously painted surface to produce a decorative effect.

Surfacer.—A pigmented composition for filling minor irregularities to obtain a smooth uniform surface preparatory to applying finish coats; usually applied over a primer and sandpapered for smoothness.

Subcommittee VII on Accelerated Tests for Protective Coatings (H. A. Nelson, chairman).—This subcommittee has recommended that the Tentative Method of Salt Spray Testing of Non-Ferrous Metals (B 117 - 41 T) be established as an emergency method for conducting salt spray tests on organic protective coatings with certain minor changes. This was established as an Emergency Method for Conducting Salt

Spray Tests on Organic Protective Coatings (A.S.T.M. Designation: ES - 3) under date of April 6, 1942.

The subcommittee has under consideration "Proposed Recommended Practice for Characteristics of Standard Carbon-Arc Accelerated Weathering Unit," prepared by Technical Committee X on Conditioning and Weathering, of Committee E-1.[8]

Considerable progress is reported by the subcommittee in the work of its subgroups.

Group 1 on House Paints (Robert W. Matlack, chairman) has continued its program of cooperative tests for studying laboratory weathering cycles designed for determining color or tint retention. In previous work a testing cycle had given promising results on a narrow range of tinted white bases. This year the same cycle was studied by using a broader range of six different white bases with two tints. Exposure tests were made in eleven locations to establish an average standard of outdoor failure. Correlation satisfactory for this purpose was obtained. However, the degrees of correlation obtained with accelerated tests made by eleven cooperators were not sufficient to enable the subcommittee to propose a standard accelerated testing procedure for tint retention of house paints. Group 1 also is preparing a series of methods for evaluating paint failure using pictorial standards.

Group 6 on Metal Protective Finishing Systems (J. C. Moore, chairman) continues to be occupied with a cooperative test program with paints used by Subcommittee XXIX. Group 6 is preparing emergency test methods for immersion testing and for high humidity (cook box) testing.

Subcommittee VIII on Methods of Analysis of Paint Materials (E. F. Hickson, chairman).—The subcommittee recommends that the present Method of Test for Consistency of Enamel Type Paints (D 562 - 41) be withdrawn and superseded by a proposed new Tentative Method of Test for Consistency of Exterior House Paints and Enamel-Type Paints.[9] The proposed method is generally similar to the present method except that the paddle-type rotor is used in place of the fork-type rotor.

This subcommittee recommends that Section 4 of the Tentative Methods of Sampling and Testing Aluminum Powder and Paste (D 480 - 41 T) be withdrawn and replaced by a method of test for the leafing properties of aluminum pigments, as appended hereto,[10] which gave good results in cooperative tests conducted by the subcommittee.

The subcommittee reports progress in connection with methods of test for certain extender pigments.

Subcommittee IX on Varnish (W. T. Pearce, chairman).—This subcommittee recommends that the Tentative Method of Test for Phthalic Anhydride Content of Alkyd Resin Solutions (D 563 - 40 T) which has been tentative for two years be continued in its present status because work is in progress which may result in an early revision. Progress was reported in studying methods for the determination on durability of varnishes, the measurement of color of dry varnish films, the measurement of drying time of varnishes, and the testing of alkyd resin solutions.

Subcommittee XV on Specifications for Pigments Dry and in Oil When Marketed in That Form (C. L. Crockett, chairman).—This subcommittee recommends that the following tentative specifications be adopted as standard without change: Barium Sulfate Pigments (D 602 – 41 T), Mica Pigment (D 607 – 41 T), Diatomaceous Silica Pigment (D 604 – 41 T), Aluminum Silicate Pigment (D 603 – 41 T), Lead Titanate (D 606 – 41 T).

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[8] See Editorial Note, p. 462.

[9] This revised method was accepted as tentative by the Society and appears in its latest revised form in the 1942 Book of A.S.T.M. Standards, Part II, p. 1372.

[10] These revised methods were accepted as tentative by the Society and appear in their latest revised form in the 1942 Book of A.S.T.M. Standards, Part II, p. 1345

The subcommittee also recommends that the Tentative Specifications for Magnesium Silicate Pigment (D 605 – 41 T) be adopted as standard with the following revision in scope:

Section 1.—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

> 1. These specifications cover the magnesium silicate pigments *available* commercially [known as talc] *in both fibrous and granular form.*

It has been realized for some time by many producers, and consumers that the present Specifications for Chrome Yellow, (D 211 – 41) are too loosely drawn and are not sufficiently specific in their requirements to properly cover the various types and shades of lead chromate pigments now available. The subcommittee, therefore, recommends for publication as tentative, revised Specifications for Chrome Yellow and Chrome Orange[11] to replace immediately the Standard Specifications for Chrome Yellow (D 211 – 41), which are accordingly recommended for withdrawal.

The subcommittee, recognizing that there is a need for a specification for toluidine toner, has prepared proposed Specifications for C. P. Toluidine Toner[12] and recommends that they be published as tentative.

The subcommittee has recommended an emergency alternate provision in the Standard Specifications for Basic Sulfate White Lead (D 82 – 41) modifying the requirements on the content of lead oxide which, it is estimated, will increase available production about 25 per cent. This Emergency Alternate Provision was approved by Committee E-10 on March 27, 1942, and issued under the designation EA – D 82.[1]

Subcommittee XVIII on Physical Properties of Materials (M. Rea Paul, chairman).—This subcommittee recommends for publication the proposed Tentative Method of Test for Spectral Characteristics and Color of Objects and Materials[13] which represents a revision and expansion of and is intended to replace the Standard Method of Test for Spectral Apparent Reflectivity of Paints (D 307 – 39).

Subcommittee XXV on Cellulose Ester Coatings (R. M. Carter, chairman).—This subcommittee recommends the following changes in the Standard Methods of Sampling and Testing Lacquer Solvents and Diluents (D 268 – 41)[4] and since these changes are made for convenience and manipulation and in no way interfere with the accuracy of the method, the revision is presented for immediate adoption:

Section 11.—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

> 11. (*a*) Weigh 1 to 2 g. of the sample in an ampoule by first weighing the empty ampoule, warming and filling, and then sealing-off and reweighing. Place the ampoule in a 200-ml. Erlenmeyer flask which contains approximately one and one-half times the theoretical quantity of 0.5 *N* [alcoholic] *aqueous* KOH required for complete saponification. *If the sample is insoluble in aqueous KOH, add sufficient water-soluble alcohol, free from saponifiable material, to effect solution.* Break the ampoule with a stirring rod and connect the flask to a reflux condenser. Heat the flask on a steam bath for 1 to 4 hr., depending on the ester being tested. During the heating, shake the set-up and contents frequently, taking the usual precautions to lose none of the contents. After the apparatus

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[11] These revised specifications were accepted as tentative by the Society and appear in their latest revised form in the 1942 Book of A.S.T.M. Standards, Part II, p. 1341.

[12] These specifications were accepted as tentative by the Society and appear in the 1942 Book of A.S.T.M. Standards, Part II, p. 1343.

[13] This method was accepted as tentative by the Society and appears in the 1942 Book of A.S.T.M. Standards, Part II, p. 1374.

has cooled, rinse the condenser with distilled water and add three drops of phenolphthalein to the contents of the flask as an indicator. Titrate the contents of the flask with 0.5 *N* HCl. Run two blanks *along with the sample* [with alcoholic KOH] *using aqueous KOH and the same amount of alcohol as was used with the sample* [along with the sample]. These blanks should check to the first decimal point. Report the result as percentage of ester by weight, allowing in the calculations for acidity or alkalinity as determined in Sections 9 and 10.

(*b*) *Optional Method.*—Weigh 1 to 2 g. of the sample in an ampoule by first weighing the empty ampoule, warming and filling, and then sealing-off and reweighing (Note). Place the ampoule in a pressure bottle of approximately 325-ml. capacity (the spring top magnesium-citrate bottle is satisfactory) which contains one and one-half times the theoretical quantity of 0.5 *N* [alcoholic] *aqueous* KOH required for complete saponification. *If the sample is insoluble in aqueous KOH, add sufficient water-soluble alcohol, free from saponifiable material, to effect solution.* Break the ampoule with a glass rod, subsequently washing the liquid adhering to the rod back into the bottle. After thorough agitation, heat the bottle and contents on a steam bath for 1 to 4 hr., depending on the ester being tested. After the proper heating, allow the bottle to cool, then open the bottle, and rinse the under side of the stopper and the sides of the neck with distilled water. Add three drops of the phenolphthalein solution to the contents of the flask as an indicator and titrate with 0.5 *N* HCl. Run two blanks [with alcoholic KOH] *along with the sample using aqueous KOH and the same amount of alcohol as was used with the sample* [along with the sample]. These blanks should check, on titration, to the first decimal point. Report the result as percentage of ester by weight, making allowance in the calculations for acidity or alkalinity as determined in Sections 9 and 10.

The subcommittee recommends for publication as tentative a Method of Test for Abrasion Resistance of Organic Coatings with the Air Blast Abrasion Tester.[14]

The subcommittee recommends that Committee D-1 request the Section on Thermometers, of Committee E-1, to establish specifications for thermometers to be used in the testing of low-boiling lacquer liquids by methods outlined in Standard D 268 – 41.

The subcommittee recommends that the following standard specifications which have stood for six years without revision be continued as standard: Specifications for Acetone (D 329 – 33), Benzene (D 361 – 36), Ethyl Acetate (D 302 – 33), Ethylene Glycol Monoethyl Ether (D 331 – 35), Acetate Ester of Ethylene Glycol Monoethyl Ether (D 343 – 35), Ethylene Glycol Monobutyl Ether (D 330 – 35), Toluene (D 362 – 36), Tricresyl Phosphate (D 363 – 36), Xylene (D 364 – 36), and Nitrocellulose (D 301 – 33).

Subcommittee XXIX on Painting of Structural Iron and Steel (A. J. Eickhoff, chairman).—This subcommittee is continuing its exposure tests covering an investigation of several methods of surface preparation of steel for painting which were described in the 1941 Report.[15]

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, H. E. Smith.
Vice-Chairman, E. W. Fasig.
Secretary, M. Rea Paul.

C. H. Rose was appointed Assistant Secretary to Committee D-1 on June 23, 1942.

This report has been submitted to letter ballot of the committee which consists of 222 voting members; 88 members returned their ballots, of whom 56 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. E. SMITH,
Chairman.

M. REA PAUL,
Secretary.
C. H. ROSE,
Ass't. Secretary.

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[14] This method was accepted as tentative by the Society and appears in the 1942 Book of A.S.T.M. Standards, Part II, p. 1368.

[15] *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 322 (1941).

Editorial Note

Subsequent to the annual meeting, Committee D-1 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative Specifications for:
Isopropyl Acetate (D 657 – 42 T),

New Tentative Methods of:
Evaluating Degree of Chalking of Exterior Paints of the Linseed-Oil Type (D 659 – 42 T),
Evaluating Degree of Checking of Exterior Paints of the Linseed-Oil Type (D 660 – 42 T),
Evaluating Degree of Cracking of Exterior Paints of the Linseed-Oil Type (D 661 – 42 T),
Evaluating Degree of Erosion of Exterior Paints of the Linseed-Oil Type (D 662 – 42 T),

Reversion of Standard to Tentative:
Standard Specifications for Basic Sulfate White Lead (D 82 – 41), as revised, replacing the Emergency Alternate Provisions EA – D 82, and

Tentative Revision of Standard Specifications for:
Zinc Oxide (D 79 – 41).

These recommendations were accepted by Committee E-10 on August 24, 1942, and assigned the A.S.T.M. serial designations indicated above. The new and revised tentative standards as well as the tentative revision appear in the 1942 Book of A.S.T.M. Standards, Part II, pp. 1367, 1397, 1399, 1402, 1405, 1339, and 1444.

On September 16, 1942, Committee E-10 accepted Emergency Alternate Provisions in the Standard Specifications for Mica Pigment (D 607 – 42) which were issued under the designation EA – D 607. They were published in ASTM Bulletin, No. 118, October, 1942, p. 55, and have been issued in the form of a pink sticker for attachment to the standard.

REPORT OF SUBCOMMITTEE VII ON ACCELERATED TESTS FOR PROTECTIVE COATINGS

Report of Group I on Paint Exposure Tests on Tint Retention

In 1940, Group I on Accelerated Tests for Fading (C. H. Rose, chairman), of Subcommittee VII, initiated a cooperative study to determine to what extent selected accelerated tests will foretell the fading characteristics of colored house paints. The paints were tested at eleven exterior locations and were also subjected to accelerated weathering tests by ten cooperators. Sections were cut as scheduled from the test panels during the exposure period and were returned to John Iliff for inspection. The returned panels were examined on February 23, 1942, by Messrs. John Iliff, H. A. Nelson and C. H. Rose. This examination and the conclusions which may be drawn therefrom are described in detail in this report which was prepared by Mr. Iliff.

Outdoor Exposure Tests:

The outdoor exposure tests were made in accordance with general directions sent to the cooperators on August 15, 1940, and which are summarized briefly in this report. Arrangements were made to have the exterior panels exposed during the week of September 2, 1940, and sections for inspection cut from the exposed panels after 1, 2, 3, 6, 9, 12, and 18 months.

The following eleven cooperators participated in the exterior exposure tests:

No.	Location	Cooperator	
1	Cincinnati, Ohio	Eagle-Picher Co.	F. L. Chinery
2	Washington, D. C.	National Bureau of Standards	E. F Hickson
3	Miami, Fla.	E. I. duPont deNemours and Co.	D. H. Dawson
4	Amarillo, Texas	E. I. duPont deNemours and Co.	D. H. Dawson
5	Wilmington, Del.	E. I duPont deNemours and Co.	D. H. Dawson
6	Palmerton, Pa.	New Jersey Zinc Co.	F. C. Schmutz
7	Columbus, Ohio	American Zinc Sales Co.	A. C. Eide
8	Buffalo, N. Y.	Pratt and Lambert	W. H. Lutz
9	Pittsburgh, Pa.	St. Joseph Lead Co.	H. E. Outcault
10	Sayville, N. Y.	National Lead Co.	C. H. Rose
11	Denver, Colo.	E. I. duPont deNemours and Co.	J. W. Iliff

Twelve tinted paints were tested; six gray and six blue. The paints contained six different bases of white pigments and each was selected to give various degrees of tint retention. The blue paints were tinted with Prussian blue and the gray paint with lampblack.

The gray paints are identified by the letters A to F, inclusive; the blue paints H to M, inclusive.

The panels for the exterior tests were prepared by the National Lead Co. and were distributed to the cooperators. The panels were standard 6 in. cedar siding, 24 in. long. The test paints were applied over two undercoats. All the panels were painted at the same time and each handled in the same manner throughout the painting. Each panel was back primed with two coats of paint.

The panels in these exposure tests were placed in a vertical position facing approximately south at the eleven test locations in the United States and fastened down in such a way that at intervals the panels could be removed and sections $2\frac{1}{2}$ in. in width cut off by sawing the panel crosswise. Before cutting a section to be removed it was marked on the face with pencil with the proper identification marks and the date of cutting. After cutting, the sawed edge of the test panel was coated with paint.

The cooperators were not required to inspect the panels. However, if at a particular time any section was sawed off it was found not to be representative of the rest of the panel this was indicated by pencil marks on the face of the section and a report made to Mr. Iliff, to whom the sections were forwarded for inspection.

Accelerated Weathering Tests:

The following ten cooperators participated in the accelerated weathering tests:

No.	Name	Company	Type of Machine
12	E. J. Ritchie	Eagle-Picher Lead Co.	N
13	C. G. Ollinger	National Carbon Co., Inc.	N
14	D. H. Dawson	E. I duPont deNemours and Co.	N
15	E. F. Hickson	National Bureau of Standards	N
16	J. W. Iliff	E I. duPont deNemours and Co.	N
17	C. H. Rose	National Lead Co.	A
18	J. H. Calbeck	American Zinc Sales Co.	A
19	G. G. Sward	National Paint, Varnish and Lacquer Assn.	Special
20	C. W. Jameson	Atlas Electric Devices Co.	A
21	R. Titherington	Passive Defense Project	N

N—National Carbon Co. Apparatus.
A—Atlas Electric Device Co. Apparatus.

The same twelve paints were tested in the accelerated weathering tests. The gray paints were identified by the letters A to F, inclusive; the blue paints H to M, inclusive.

Each cooperator prepared the panels for his test. The panels were made of wood, preferably cedar, and of a size and type regularly used by each respective cooperator. The panels were painted front, back and edges with two coats of paint, one coat primer, one coat test paint. A total of thirty-six panels were prepared by each cooperator, three with each paint.

TABLE I.—MODIFIED ACCELERATED WEATHERING MACHINE SCHEDULE.[a]

			Hours of Light
Monday	9:00 A.M.......	Trim lamp	8
	5:00 P.M.......	Trim lamp	Approx. 10
Tuesday	9:00 A.M.......	Ice water bath for 2 hr.	
	11:00 A.M.......	Inspection	
	5:00 P.M.......	Trim lamp	Approx. 10
Wednesday	9:00 A.M.......	Trim lamp	8
	5:00 P.M.......	Trim lamp	Approx. 10
Thursday	9:00 A.M.	Trim lamp	8
	5:00 P.M.......	Trim lamp	Approx. 10
Friday	9:00 A.M.......	Ice water bath for 2 hr.	
	11:00 A.M.......	Inspection	
	5:00 P.M.......	Trim lamp	Approx. 10

[a] This schedule for National Carbon Co. machines. Other machines were run in the manner usually used by the respective cooperators with ice water bath, and inspection and rest periods as in the above schedule.

Recommended conditions for operating the National X-1A accelerated weathering unit:

The arc to be operated at 60 amp. at 50 v., using Sunshine carbons, with Corex D filters.

The unit to be operated with one water spray, and an uncovered drum. The panel carriage to make one rotation in 2 hr.

Panels are changed from top to bottom rack (or *vice versa*) at each inspection to equalize for any differences in temperature or radiated energy between the two positions.

The priming coat was allowed to dry for one week before repainting. The finish coat was allowed to dry for one week before testing.

The panels were exposed to the conditions of an accelerated weathering similar to that shown in Table I. This schedule applied to the National Carbon Co.

Machine. Cooperators using other machines selected their own schedule but were required to include an ice water bath, inspection and rest periods as called for in the schedule in Table I.

The test panels were cut into thirds as the exposure test progressed so that a third of a panel could be removed from the test each week. The sawed edge of the panel returned for test was given a coat of spar varnish.

The cooperators were not required to inspect the panels. However, if at a particular time any section was sawed off it was found not to be representative of the rest of the panel, this was indicated by pencil marks on the face of the section and a report made to Mr. Iliff, to whom the sections were forwarded for inspection.

TABLE II.—ORDER OF FADING AT EXTERIOR TEST LOCATIONS, AND NUMBER OF MONTHS FOR FIRST FADING BREAK[a]

Cooperator	1	2	3	4	5	6	7	8	9	10	11
Order of Failure	Cincinnati, Ohio	Washington, D.C.	Miami, Fla.[b]	Amarillo, Texas[b]	Wilmington, Del.	Palmerton, Pa.	Columbus, Ohio	Buffalo, N. Y.	Pittsburgh Pa.[c]	Sayville, N.Y.	Denver, Colo.[b]
Tinted Gray Paints:											
1 (least)	A 12+	A 12+	B 9−	B	A 9+	A 12	A 12+	A 12	D 9	A 12	B 12+
2	F 9	F 12	E 9−	E	F 9	F 9+	F 9	F 9	A 6	F 12	E 12+
3	B 9	B 12	A 6	C	B 9	E 9	E 9	E 9	F 6	B 12−	C 12
4	E 9	E 12	F 6	F	E 9	B 9	C 9	C 9	C 6	E 12−	F 12
5	D 9	C 12−	C 6−	A	C 9	C 9	D 9	B 9	E 6	C 9+	D 9
6 (most)	C 6+	D 9	D 6−	D	D 9	D 9	B 9	D 9−	B 6	D 9+	A 9
Tinted Blue Paints:											
1 (least)	M 9	M 9	J 6−	J 6	K 6	M 9	K 9	M 9	K 9	M 6	M 6
2	J 9	K 9	K 6−	M 6	J 6	K 9	J 9	K 9	J 9	J 6	J 6
3	K 9	J 9	M 6−	K 6	I 6	J 9	M 9−	J 9	H 9	K 6	K 6
4	H 6+	I 9−	H 3−	H 6	M 6	I 9	I 9−	H 9−	I 9	H 6	H 6
5	I 6+	H 6+	I 2−	I 6	H 6	H 9−	H 6	I 6+	M 9−	I 6	I 6
6 (most)	L 2	L 1	L 1−	L 1−	L 1−	L 1	L 1−	L 1	L 1	L 1−	L 1−

[a] The numbers which follow the letters designating the different paints indicate the month after beginning of the test at which the first break in fading of that paint was noted.

[b] Climate not typical of that of more populated areas.

[c] Strong hydrogen sulfide gas attack evident, held up chalking. In the case of the gray paints gas attack was evident at 6 months.

Summary of Test Results:

The method used for an evaluation of the accelerated weathering results in comparison with the exterior exposure performance was as follows:

The first step was to set up a "standard" for outdoor performance.

Reports were received from the eleven exterior exposure stations up to and including the 12 month's exposure period. Only a portion of the eighteenth month's exposure sections were received and these were examined to a sufficient extent to show that no reversal of conclusions would be caused through the use of this additional information.

The sections from each of the panels at the exposure stations over the 12 month period were arranged according to paint and in proper chronological order. Thus a group of sections was available over the 12 month period for each paint and from each exposure station.

The order of failure at each exposure station was established by arranging the groups of sections from the test panels. The results of this arrangement are shown in Table II.

It will be seen that quite close agreement was obtained between seven of the eleven exposure stations. All of these exposure stations, namely, Cincinnati, Ohio; Washington, D. C.; Wilmington, Del.; Palmerton, Pa.; Columbus, Ohio;

Buffalo, N. Y.; and Sayville, N. Y.; are in the part of the country where the heaviest population concentration exists and where the climate may be considered comparable or "average." Miami, Fla.; Amarillo, Tex.; and Denver, Colo., did not give as close duplication as the other seven locations. This is to be expected because the climate in these three places is considerably different from that of the Middle West and Eastern seaboard represented by the seven "average" locations mentioned. The fourth exception is that of Pittsburgh, Pa. While located within the area covered by the seven "standard" stations, so much hydrogen sulfide darkening was apparently obtained with some of the paints that the results were upset. Consequently, it was also excluded in establishing a standard.

TABLE III.—COMPARISON OF "AVERAGE STANDARD" IN OUTDOOR EXPOSURE TESTS WITH RESULTS OF ACCELERATED WEATHERING TESTS, AND NUMBER OF WEEKS FOR FIRST FADING BREAK.[a]

Cooperator		12	13	14	15	16	17	18	19	20		21
Average "Standard"	Average "Position of Standard"									ARVBW	ARVTA	
Tinted Gray Paints:												
A	1	E 3	E 3	E 4	C 2	E 3	F 5	E 7	A and C 1	A 6	E 2	B 3
F	2	B 3	C 3	B 4	B 2	B 3	B 5	B 5	eroded 1	C 5	B 1+	F 3
E	3.6	F 3	F 3	C 3+	E 2	C 3	E 5	C 4+	through 1	E 5	C 1	C 2
B	3.9	C 3	B 3	B 3	F 2	F 2	C 4	F 4+	1	B 4	D 1	F 2
C	4.9	D 2	D 3	F 3	D 2	D 2	D 3	D 4	Too fast 1	F 4	F 1	D 2
D	5.7	A 2−	A 3−	A 2	A 2−	A 2	A 3	A 3+	to rate 1−	D 3	A 1−	A 2−
Tinted Blue Paints:												
M	1.6	K 3	K 3	J 4−	K 2	K 3	K 4	K 5	Too fast	K 4+	J 2−	J
K	2.0	J 3	J 3	K 4−	H 2	J 3	H 4	J 4+	to rate	J 4	K 1+	K 3
J	2.4	H 3−	H 3−	H 4−	J 1	H 3	M 3	H 4		H 4	H 1	M 3
H	4.6	M 2+	M 2+	M 3	M 1	M 3	J 3	M 3		M 4	M 1	H 3
I	4.6	I 2	I 2	I 3	I 1	I 2+	I 3	I 3		L 3	I 1	I 3
L	6	L 1−	L 1	L 2	L 1	L 2	L 1	L 1−		I 3	L 1−	L 2

[a] The numbers which follow the letters designating the different paints indicate the number of weeks from beginning of the test at which the first break in fading of that paint was noted.

The standard order of paint performance is shown in the left-hand column of Table III. This order was established by averaging the results of the seven "average" stations. The results of this average are shown in column two, "average position of standard," which shows a relatively well established position of paint items.

For the sake of verification, the month of the first break in fading is also included in Table II.

The second step was to determine the relative performance of the accelerated tests and the "standards."

The accelerated weathering results from each of the cooperators was also arranged in the same manner as shown in Table III. Similarly, the time in weeks for the first break in fading was also included as a verification of this established order of failure.

It will be seen that fairly close agreement between individual accelerated results was obtained with most of the cooperators. The exception to this was a special cycle by cooperator 20 with the heading "20 ARVBW" in Table III.

It is obvious from a comparison with the so-called "standard" of the accelerated test results, particularly in the case of the gray paints, that no agreement between the accelerated weathering fading results and exterior exposure was obtained. Second, the rate of accelererated weathering failure for fading purposes was probably entirely too fast in practically all of the cycles used. Whether a slower cycle might give more favorable results is, of course, unknown. The special cycle of cooperator 20 was closer in agreement with respect to the gray paints than any of the others in that paints A and D were put in the proper position, and paints E and B remained in the middle, but paints C and F were reversed. On the other hand, this cycle was relatively worse than the other cycles with respect to the blue paint.

Conclusions:

The following conclusions may be drawn from these tests:

1. None of the accelerated weathering cycles used in these tests can be used to predict fading performance of house paints with various white base compositions.

2. All of the accelerated weathering cycles tested were too severe and the rate of failure was so rapid that a distinction between the performance of individual paints was often not picked up between weekly examinations of the exposure panels.

Respectfully submitted on behalf of the subcommittee,

H. A. NELSON,
Chairman.

REPORT OF COMMITTEE D-2

ON

PETROLEUM PRODUCTS AND LUBRICANTS

Committee D-2 on Petroleum Products and Lubricants held three regular meetings during the past year (in June at Chicago, Ill., in January at Detroit, Mich., and in March at Cleveland, Ohio).

During the year, Section VI on Acid Heat, of Technical Committee A on Gasoline, has been discontinued, and a special subcommittee on coordination of test methods has been organized.

Table I shows the changes in status during the year of American standards for petroleum products, approved by the American Standards Association on the recommendation of Sectional Committee Z11 on Petroleum Products and Lubricants.

The voting membership of Committee D-2 now consists of 36 producers, 25 consumers, and 12 general interest members.

Subcommittee III on Paraffin Wax (L. C. Beard, Jr., chairman) has conducted cooperative tests on a new Method of Test for Oil Content of Paraffin Wax and recommends that the method which appears in Appendix I be published as information only. The results of the cooperative tests are shown in Table II.

Subcommittee VI on Color (H. M. Hancock, chairman) has prepared a Proposed Method of Test for Color of Lubricating Oil by Means of Photoelectric Colorimeter, appearing in Appendix II, which is presented for publication as information. Studies made by the subcommittee in the preparation of this method are briefly discussed in its report appended hereto.

Subcommittee XIII on Neutralization Number and Saponification (H. P. Ferguson, chairman) has continued its studies of methods for neutralization number and is recommending that the two methods published as information in the 1941 report,[1] be published with minor revisions as tentative standards. These methods as appended hereto[2] replace the present Tentative Methods

TABLE I.

Title	ASTM Designation	ASA Number
NEW AMERICAN STANDARDS		
Test for Distillation of Plant Spray Oils	D 447 - 41	Z11.43—1941
Test for Vapor Pressure of Petroleum Products (Reid Method)	D 323 - 41	Z11.44—1941
Method for Calculating Viscosity Index	D 567 - 41	Z11.45—1941
Method for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity	D 446 - 39	Z11.46—1941
REVISIONS OF AMERICAN STANDARDS		
Test for Carbon Residue of Petroleum Products (Conradson Carbon Residue)	D 189 - 41	Z11.25—1941
Test for Distillation of Crude Petroleum	D 285 - 41	Z11.32—1941
Test for Distillation of Gas Oil, and Similar Distillate Fuel Oils	D 158 - 41	Z11.26—1941
WITHDRAWAL OF APPROVAL AS AMERICAN STANDARD		
Method of Test for Knock Characteristics of Motor Fuels	D 357 - 40	Z11.37—1940

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 41, pp. 342, 348 (1941).

[2] These methods were accepted as tentative by the Society and appear in the 1942 Book of A.S.T.M. Standards, Part III, p. 954 and 958.

of Test for Neutralization Number of Petroleum Products and Lubricants (D 188 – 41 T) which are to be withdrawn.

The subcommittee also completed a revision of the Tentative Method of Test for Saponification Number (D 94 – 41 T) that includes a statement on reproducibility of the method as applied to fats and fatty oils, based upon cooperative work summarized in Table III. The revised method is appended hereto.[3]

TABLE II.—OIL CONTENT OF PARAFFIN WAX.

Laboratory	Oil Content, per cent		
	Crude Scale Wax	Refined Wax	De-oiled Wax plus Approximately 1 per cent Pressed Distillate
A	3.61	0.67	1.14
	3.58	0.60	1.10
B	3.95	0.60	1.24
	3.81	0.65	1.18
C	4.14	0.99[a]	1.30
	4.09	0.94[a]	1.35
D	3.90	0.74	1.90[a]
	4.01	0.66	1.79[a]
E	4.26[a]	0.79	1.31
	4.34[a]	0.86	1.47
F	3.67	0.51	1.48
	3.81	0.56	1.43
G	3.71	0.69	1.22
	3.67	0.73	1.07
Average	3.83	0.67	1.27
Avg. Deviation from Average	0.16	0.08	0.12
Max. Deviation from Average	0.31	0.19	0.21

[a] Not used in calculating averages.

Technical Committee A on Gasoline (C. B. Veal, chairman) is proposing, in addition to several recommendations on standards covered later in this report, that the Proposed Method of Test for Potential Gum in Aviation Gasoline, published as information in the 1941 report,[4] be revised editorially and published again as information as it appears in Appendix III.

Technical Committee B on Lubricants (H. C. Mougey, chairman) is recommending that the Proposed Method of Test for Oxidation Characteristics of Heavy-Duty Crankcase Oils appearing in Appendix IV be published as information. This method involves the use of a Chevrolet engine with suitable loading and accessory equipment. The experimental background is covered in two papers by H. C. Mougey and J. A. Moller.[5]

Technical Committee C on Turbine Oils (F. C. Linn, chairman) developed the proposed Tentative Method of Test for Rust-Preventing Characteristics of Steam Turbine Oil in the Presence of Water.[6] The report of this subcommittee is appended hereto.

TABLE III.—SUMMARY OF COOPERATIVE TESTS ON SAPONIFICATION NUMBERS OF FATS AND FATTY OILS.

Laboratory	Degras	Blown Rapeseed Oil	No. 1 Lard	Soybean Oil	Cotton Seed Oil	Sperm Oil	Sulfur Saponifiable[b]
A	118.0	211.5	193.0[a]	192.5	191.0[a]	141.3	144.6
B	116.3	212.9	190.4	192.0	188.2	140.9	.....
C	113.6	209.0	190.7	195.0[a]	189.3	136.0	147.3
D	117.2	196.4[a]	189.5	188.7[a]	185.8	135.6	166.7[a]
E	112.0	211.8	189.4	190.4	186.6	137.7	145.4
F	.....	.....	191.3	193.2	186.4	139.6	.....
G	114.1	215.9	191.1	192.7	185.7	136.7	148.5
H	112.0	211.7	191.5	190.6	187.2	137.6	145.5
Average	114.7	212.1	190.6	191.9	187.0	138.2	146.3
Avg. Deviation	2.1	1.5	0.7	0.9	1.0	1.8	1.3
Max. Deviation	3.3	3.8	1.2	1.5	2.3	3.1	2.2

[a] Results omitted from average.

[b] Fatty oils which have been saponified with sulfur. Materials of this type are being used as additives in crankcase oils, and the committee desired to determine whether reproducible results could be obtained on oils containing these additives. These products are, however, excluded from the scope of the revised Tentative Method of Test for Saponification Number of Petroleum Products by Color-Indicator Titration (D 94 – 42 T.).[3]

I. NEW TENTATIVE STANDARDS

The committee recommends that the following four methods be accepted for publication as tentative:

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[3] This revised method was accepted as tentative by the Society and appears in its latest revised form in the 1942 Book of A.S.T.M. Standards, Part III, p. 972.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 351 (1941).

[5] H. C. Mougey and J. A. Moller, "The Testing of Heavy-Duty Motor Oils," *Proceedings*, Am. Petroleum Inst., Vol. 22, Part III, pp. 100 to 121 (1941); and Soc. Automotive Engrs., Annual Meeting, January, 1942.

[6] This method was accepted as tentative by the Society and appears in the 1942 Book of A.S.T.M. Standards, Part III, p. 969.

Test for Neutralization Number of Petroleum Products by Color-Indicator Titration,[2] largely as published as information in the 1941 report of the committee,[1]

Test for Neutralization Number of Petroleum Products by Electrometric Titration,[2] essentially as published as information in the 1941 report of the committee,[1]

Test for Rust-Preventing Characteristics of Steam-Turbine Oil in Presence of the Water,[6] and

Method for Conversion of Kinematic Viscosity to Saybolt Furol Viscosity,[7] as published for information in the 1941 report of the committee.[8]

II. REVISIONS OF TENTATIVE STANDARDS

The committee recommends revision of five tentative standards as follows:

Tentative Method of Test for Gum Stability of Gasoline (D 525 – 41 T):[9]

The following minor changes are recommended by Technical Committee A on Gasoline (C. B. Veal, chairman):

Title.—Change "gum" to read "oxidation."

Section 1.—In the first sentence omit the word "gum."

Note 4.—Change from its present form to read as follows:

NOTE 4.—For convenience in handling, it is suggested that a tire valve and stem be attached to a three-way pressure cock which in turn is attached to the needle valve and that oxygen be introduced through a suitable high-pressure air hose and fitting. It is considered safer practice to release the gas in the bomb through the third unobstructed port of the three-way cock rather than through the small orifice of the tire valve. The needle valve must not be eliminated, as it is necessary to insure against leaks at this point during the test.

Section 2 (*d*).—Change the first sentence from its present form: namely,

A 200-psi. maximum pressure gage of either the indicating or recording type shall be used having a precision of not more than 50 psi. per inch of travel of the pointers along the arc of the scale throughout the range of 100 to 180 psi. and having divisions on the chart or scale not greater than 5 psi.

to read as follows:

A pressure gage with a maximum reading of 200 psi. shall be used. It may be of either the indicating or the recording type. The scale for the range 100 to 180 psi. shall average at least 1 inch per 50-lb. pressure differential, measured along the arc of the scale. Intervals of division shall be 5 psi. or less. The accuracy shall be 1 per cent of the total scale interval.

Section 3 (*b*).—Change the last sentence from its present form: namely,

Close the bomb (Note 6), introduce oxygen until a pressure of 125 psi. is obtained, and then, after 10 ± 2 min., adjust the pressure to between 100 to 102 psi.

to read as follows:

Close the bomb and introduce oxygen until a pressure of about 100 to 102 psi. is obtained. Allow the gas in the bomb to escape in order to flush out the air originally present. Introduce oxygen again until a pressure of 100 to 102 psi. is obtained and observe for leaks. If there are no leaks, the test may be continued.

Note 6.—Change from its present form to read as follows:

NOTE 6.—An initial rapid drop in pressure, generally not over 6 psi., may be observed because of the solution of oxygen in the sample. If, after a few minutes, the rate of pressure drop does not exceed 1 psi. in 10 min., the absence of leaks may be assumed.

Section 4.—Correct the definition of IP_t to read as follows by the addition of the italicized words:

IP_t = ***Induction period at the*** average temperature of the test.

Tentative Method of Test for Knock Characteristics of Aviation Fuels (D 614 – 41 T):[10]

The following minor changes are recommended by Technical Committee A on Gasoline (C. B. Veal, chairman), largely to bring the method into conformity where possible with the proposed revision of the Tentative Method of Test for Knock Characteristics of Motor Fuels (D 357 - 41 T):

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[7] This method was accepted as tentative by the Society and appears in the 1942 Book of A.S.T.M. Standards, Part III, p. 982.

[8] *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 341 (1941)

[9] 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 269.

[10] 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 292.

Footnote 2.—Change the second sentence of the second paragraph to read as follows by the addition of the italicized words and the omission of those in brackets:

> Based on tests of a variety of aviation fuels in current aircraft engines and on service experience, this method [satisfactorily] rates such fuels [for purposes of procurement and use under normal operating conditions] ***under conditions approximating best power, but is not intended to correlate with full-rich take-off conditions.***

Section 4 (b).—In the first sentence, delete the word "leaded" in the phrase "leaded secondary reference fuels;" also delete the word "unleaded" in footnote 5.

Section 5 (c).—Omit this paragraph, relettering the subsequent paragraphs accordingly.

Section 5 (g).—Reletter as Paragraph (h) and add a new paragraph to read as follows:

> (*g*) *Adjustment of Micrometer.*—When the clearance volume to the top face of the thermal-plug hole is 140 ± 0.5 ml., as measured by the "tilt" method (see Appendix), with the piston at top dead center, the micrometer shall be set to read 0.500 in.

Section 5 (i).—In the second sentence add the reference "(Appendix)" after the words "compression ratio."

Add the following as Note 1 at the end of the first paragraph, with reference at the end of the first sentence:

> NOTE 1.—For routine purposes a blend of certified secondary reference fuels may be used instead of the blend of primary reference fuels.

Section 5 (k).—Change the requirements for the breaker-point gap from "0.015 in." to read "0.020 in."

Section 5 (m).—Change from its present form to read as follows:

> (*m*) *Valve Clearances.*—Hot and running clearances shall be 0.008 in. intake and 0.006 in. exhaust with the engine running under standard operating conditions on a reference fuel of 87 octane number.
>
> NOTE 2.—The valves may be adjusted with the engine stopped, either hot or cold, if appropriate allowances are made so that the hot and running clearances will be as specified.

Section 5 (q).—At the end of this paragraph add the reference "See Section A5 (*a*)" and add a new paragraph to read as follows:

> When air of the above humidity prevails in the engine room through the operation of natural factors or the use of air conditioning, it may be used without further conditioning. Its humidity shall be determined by use of the type of sling psychrometer currently approved by the U. S. Weather Bureau.

Section 12.—Add a new section on reproducibility of results identical with Section 14 of proposed revised Method D 357 to read as follows:

> 12. *Reproducibility of Results.*—Results obtained by this procedure with different C.F.R. engines and in different laboratories should differ by not more than two octane numbers. Differences as small as 0.2 octane number between two test samples of similar characteristics, may be detected by direct comparison using the procedure described in Section 10.

Section A1.—Change the limitation on atmospheric pressures to read "not less than 26.0 in. (660 mm.) of mercury."

Section A2.—Insert the word "minimum" after "exhaust pipe."

Section A4 (e).—Add the following note at the end of this section:

> NOTE.—The recommended method consists of the following steps:
>
> 1. Remove the intake and exhaust push rods.
> 2. Swing the intake and exhaust rocker arms to a vertical position.
> 3. Mount a dial indicator on the rocker-arm support bracket with the indicating plunger directly over and pointing at the intake tappet.
> 4. Place a piece of drill rod between the intake tappet and the indicating plunger. This rod should be approximately ½ in. in diameter, have a spherical end where it

rests in the tappet and an indentation on the other end in which the indicating plunger will rest.

5. Set dial gage position so its plunger is approximately in the middle of its travel when the tappet is on the base circle of the cam. Then set the dial indicator at zero.
6. Rotate the flywheel in its running direction until the indicator shows that the cam is lifting the tappet. Mark the flywheel directly under the t.d.c. pointer at the angle where the tappet has been lifted 0.010 in. Repeat this operation two or three times to make sure of the angle at which this 0.010-in. lift is obtained. This is the point at which the effective valve opening starts. The part of the cam below the 0.010-in. lift is the quieting ramp and does not contribute to the effective valve opening.
7. This mark should be at the timing line on the flywheel marked "I.O. 0.010" which is 10 deg. a.t.d.c.
8. If the cam timing is not within $\frac{3}{8}$ in. of the mark on the flywheel, the camshaft will need retiming either by shifting the cam gear with respect to the crankshaft gear or by relocating the cam gear on its shaft by using one of the other two keyways provided. Shifting the timing one full gear tooth will make 1.48-in. change on the flywheel. The extra keyways provided on the camshaft permit adjusting the timing in 0.37 in. and 0.74 in. increments.

Section A4 (f).—Change to read as follows:

(*f*) *Valve Clearances.*—Hot and running clearances shall be 0.008 in. intake and 0.006 in. exhaust with the engine running under standard operating conditions on a reference fuel of 87 octane number. To avoid possible change in clearances throughout the compression-ratio range, set the top linkage of the rocker-arm carrier and the rocker arms in a horizontal plane at 0.500-in. micrometer setting with valves closed.

Section A4 (i).—Insert new paragraph as follows:

Micrometer.—A suitably mounted micrometer for measuring the height of the cylinder with respect to the crankcase.

Section A6 (c).—Change the spark plug type to RJ-11-64A.

Section A7.—Change to "A7 (a)" and add a new Paragraph (*b*) reading as follows:

(*b*) Ethylene glycol shall be used as coolant. A maximum of 1 per cent by weight or by volume of a suitable corrosion inhibitor may be added to the ethylene glycol.[a]

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[a] Suggested corrosion inhibitors are sodium nitrite or triethanolamine phosphate.

Section A13(e).—Delete the second sentence.

Appendix.—Insert a new Appendix identical with that being added in the proposed revision of Tentative Method D 357 - 41 T.

Tentative Method of Test for Knock Characteristics of Motor Fuels (D 357 - 41 T)[11]

A revision of this method involving the addition of knock-intensity guide tables and editorial rearrangement is recommended by Technical Committee A on Gasoline (C. B. Veal, chairman). The revised method is appended hereto.[12]

Tentative Method of Test for Kinematic Viscosity (D 445 - 39 T):[13]

The following changes are recommended by Subcommittee V on Viscosity (J. C. Geniesse, chairman):

Section 2.—At the end of the section add the following four sentences and accompanying footnote:

Various types of apparatus have been used. Three types are shown in the Appendix. Others are satisfactory. For very dark or black oils it may be necessary to use the reversed-flow type.[a] Some operators use the latter type for both light and dark oils.

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[a] M. R. Cannon and M. R. Fenske, "Viscosity Measurement," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 13, p. 299 (1941).

Fig. 1.—Substitute a new sketch for the Ubbelohde viscosimeter with one capillary tube to portray the position of the various bulbs and tubes more accurately, as shown in the accompanying Fig. 1.

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[11] 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 281.

[12] This revised method was accepted as tentative by the Society and appears in the 1942 Book of A.S.T.M. Standards, Part III, p. 935.

[13] 1939 Book of A.S.T.M. Standards, Part III, p. 647.

Table I.—Change the inside diameters of tubes 1, 2, 3, and 4 to read as follows:

			Ubbelohde Viscosimeter	Fitz-Simons' Modification
Inside diameter of	tube 1, mm.		8 to 10	8 to 12
	tube 2, mm.		3 to 6	4 to 6
	tube 3	series 1 to 3, mm.	3 to 5	4 to 6
		series 4 and 5, mm.	4.5 to 6.5[a]	4 to 6
	tube 4	series 1 to 3, mm.	3 to 5	4 to 8
		series 4, mm.	4.5 to 6.5	4 to 8
		series 5, mm.	7 to 10	4 to 8

[a] These dimensional limits are important only for a few millimeters of the tube adjacent to its junction with tube 5.

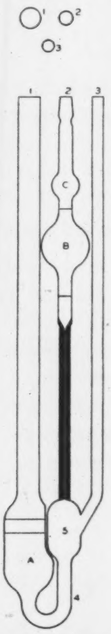

FIG. 1.—Ubbelohde Viscosimeter with One Capillary Tube.

This rearrangement corrects an error in the diameter of tube 4, and specifies a larger diameter for tube 3, series 4 and 5, of the Ubbelohde viscosimeter to prevent blocking off this tube when oil of high viscosity is being tested.

Add the following supplementary dimensions for convenience in making replacements:

Supplementary dimensions (not mandatory) to insure fitting the majority of holders now in use:

Outside diameter of tube 1, mm.	11 ± 0.2
Outside diameter (approximate) of tube 2, mm.	7
Outside diameter (approximate) of tube 3, mm.	6

Tubes 1, 2, and 3 shall be spaced so they will fit loosely in a template made as follows:

Lay off an isosceles triangle with the base 18.0 mm. in length and the apex 16.5 mm. from each end of the base. Drill a $\frac{29}{64}$-in. hole (for tube 1) centered with the right end of the base, a $\frac{3}{8}$-in. hole (for tube 2) centered with the left end of the base, and a $\frac{5}{16}$-in. hole (for tube 3) centered with the apex of the triangle. Make an identifying mark on the top surface of the template to insure against its being applied upside down.

Table II.—Add the dimensions for capillary tube No. 5 of the Ubbelohde Viscosimeter as follows:

Capillary Diameter, mm.	Approximate Calibration Constant, C^a	Approximate Viscosity Range	
		Centistokes	Saybolt Universal Seconds
6.1 to 6.3	100	8 000 to 100 000	37 000 to 460 000

Table III.—Change the inside diameter of tube 2 by the addition of the italicized words and figures to read as follows:

Inside diameter	of tube 2, *50 to 300 series*, mm.	1.5 to 2.5
	of tube 2, 400 and 500 series, mm.	*2.5 to 3.5*

This proposed change is to prevent blocking off the tubes with oils of high viscosity.

Add the following dimension to the table for convenience in making replacements:

Distance between center lines of tubes 1 and 2,[a] mm.	25 ± 2

[a] This dimension is not mandatory. It is included as a guide in making replacements in a holder to which it applies.

Tentative Method of Test for Saponification Number (D 94 – 41 T):[14]

Subcommittee XIII on Neutralization Number and Saponification (H. P.

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[14] 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 305.

Ferguson, chairman) recommends changes in the title and in the statement of scope of this method, also the addition of a definition and a brief summary of the principle of the method, together with other changes referred to earlier in this report. The revised method is appended hereto.[3]

III. REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions in four standard methods of test, as set forth below, and accordingly asks for a nine-tenths affirmative vote at the annual meeting in order that these modifications may be referred to letter ballot of the Society:

Standard Method of Test for Gum Content of Gasoline (D 381 – 36):[15]

The following minor change is recommended by Technical Committee A on Gasoline (C. B. Veal, chairman):

Section 3 (a).—Change the fourth sentence from its present form: namely,

> If the air in the balance to be used is dried with a desiccant then this same desiccant shall be used in the desiccator; otherwise no drying agent shall be used in the desiccator.

to read as follows:

> Do not use a drying agent in either the balance case or in the desiccator.

Standard Method of Test for Flash Point by Means of the Pensky-Martens Closed Tester (D 93 – 40):[16]

The following minor change is recommended by Subcommittee XII on Flash Point (J. B. Terry, chairman):

Section 2.—At the end of Paragraph (*a*) add a sentence to read as follows:

> There may be a fillet approximately $\frac{5}{32}$ in. (0.397 cm.) in radius inside the bottom of the cup.

This fillet facilitates cleaning the cup.

Standard Method of Test for Melting Point of Paraffin Wax (D 87 – 37):[17]

The following change is recommended by Subcommittee III on Paraffin Wax (L. C. Beard, Jr., chairman) to make the method applicable to the determination of melting point of waxes that do not give four consecutive 30-sec. readings within a range of 0.2 F.:

Section 4 (e).—Change the third sentence to read as follows by the addition of the italicized number and the omission of the number in brackets:

> The melting point thermometer reading, estimated to 0.1 F. shall be observed and recorded every [30] *15* sec.

Section 4 (f).—Change the first and second sentences to read as follows by the addition of the italicized number and word and the omission of those in brackets:

> The melting point thermometer reading, estimated to 0.1 F., shall be observed and recorded every [30] *15* sec., for at least 3 min. after the temperature again begins to fall after remaining almost constant. The record of temperature readings shall then be inspected and the average of the first [four] *five* readings that lie within a range of 0.2 F. shall be considered as the uncorrected melting point.

Standard Method of Test for Vapor Pressure of Petroleum Products (Reid Method) (D 323 – 41):[18]

The following changes are recommended by Subcommittee XXII on Natural Gasoline (G. G. Oberfell, chairman) to clarify the description of the apparatus and to provide against the possibility of error when the air chamber and gasoline chamber are connected:

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[15] 1939 Book of A.S.T.M. Standards, Part III, p. 167.

[16] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 47.

[17] 1939 Book of A.S.T.M. Standards, Part III, p. 180.

[18] 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 24.

Section 2 (a), (b), (c), and (d).—Change from the present form to read as follows:

2 (*a*) *Air Chamber.*—The upper section or air chamber, shown in Fig. 1 (*c*) (the accompanying Fig. 2) shall be a cylindrical vessel $2\frac{1}{16} \pm \frac{1}{16}$ in. in diameter and $10 \pm \frac{1}{8}$ in. in length, inside dimensions, with the inner surfaces of the ends slightly sloped to provide complete drainage from either end when held in a vertical position. On one end of the air chamber, a suitable gage coupling with internal diameter not less than $\frac{3}{16}$ in. shall be provided to receive the $\frac{1}{4}$-in. gage connection. In the other end of the air chamber an opening approximately $\frac{1}{2}$ in. in diameter shall be provided for coupling with the gasoline chamber. Care shall be taken that the connections to the end openings do not prevent the chamber from draining completely.

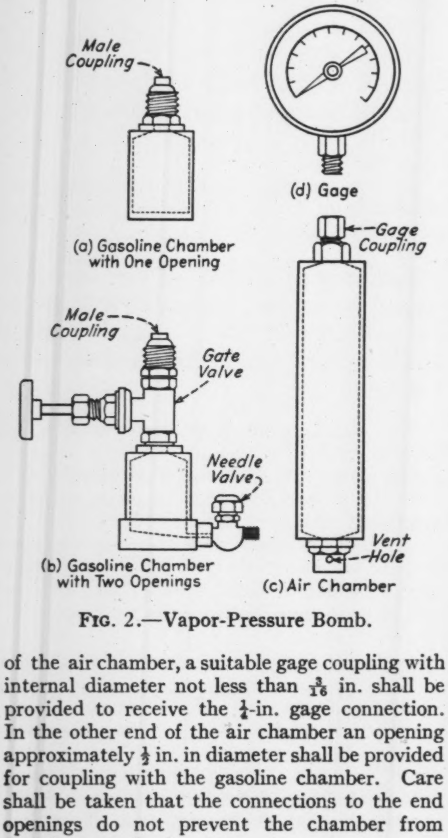

FIG. 2.—Vapor-Pressure Bomb.

(*b*) *Gasoline Chamber.*—The lower section, or gasoline chamber, shown in Fig. 1 (a), shall be a cylindrical vessel of the same inside diameter as the air chamber and of such volume as to fulfill the requirements of Paragraph (*e*). In one end of the gasoline chamber an opening approximately $\frac{1}{2}$ in. in diameter shall be provided for coupling with the air chamber. The inner surface of the end containing the coupling member shall be sloped to provide complete drainage when inverted. The other end of the gasoline chamber shall be completely closed.

(*c*) *Gasoline Chamber for Sampling from Closed Vessels.*—For this use the lower section, or gasoline chamber, shown in Fig. 1 (b), shall be essentially the same as described in Paragraph (*b*) except that a $\frac{1}{4}$-in. needle valve shall be attached near the bottom of the gasoline chamber and a $\frac{1}{2}$-in. gate valve shall be introduced in the coupling between the chambers. The volume of the gasoline chamber including only the capacity enclosed by the valves shall fulfill the requirements of Paragraph (*e*).

(*d*) *Method of Coupling Air and Gasoline Chambers.*—Any method of coupling the air and gasoline chambers may be employed, provided that no gasoline is lost during the coupling operation, that no compression effect is caused by the act of coupling, and that the assembly is free from leaks under the conditions of test. To avoid displacement of gasoline during assembly, it is desirable that the male fitting of a suitable coupling be on the gasoline chamber. To avoid compression of air during the assembly of a suitable screw coupling, a vent hole may be used to insure atmospheric pressure in the air chamber at the instant of sealing.

Figure 1.—To be consistent with revised wording of Section 2 (*d*), substitute the accompanying Fig. 2 for the present Fig. 1.

Section 3 (a).—Change the first sentence to read as follows by the addition of the italicized words and figures:

The pressure gage shall be a Bourdon-type spring gage of test-gage quality $4\frac{1}{2}$ to $5\frac{1}{2}$ in. in diameter *provided with a nominal $\frac{1}{4}$-in. male-thread connection* with a passageway not less than $\frac{3}{16}$ in. in diameter from the Bourdon tube to the atmosphere.

IV. ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the following three tentative standards be approved for reference to letter ballot of the Society for adoption as standard:

Tentative Method of Test for Carbon Residue of Petroleum Products (*Ramsbottom Carbon Residue*) **(D 524 – 41 T)**,[19] on the recommendation of Subcommittee XXIII on Carbon Residue (L. C. Beard, Jr., chairman),

Tentative Method of Test for Dropping Point of Lubricating Grease **(D 566 – 40 T)**,[20] on the recommendation of Subcommittee IV on Grease (F. E. Rosenstiehl, acting chairman), and

Tentative Method of Test for Tetraethyl Lead in Gasoline **(D 526 – 41 T)**[21] on the recommendation of Technical Committee A on Gasoline (C. B. Veal, chairman).

V. Withdrawal of Tentative Standard

The committee recommends the withdrawal of the Tentative Methods of Test for Neutralization Number of Petroleum Products and Lubricants (D 188 – 41 T)[22] on the recommendation of Subcommittee XIII on Neutralization Number and Saponification (H. P. Ferguson, chairman) as it is superseded by the two new proposed tentative standards.[2]

VI. Tentative Standards Continued as Tentative

The following tentative standards have remained in the tentative status for two years or longer without revision:

Test for Color of Lubricating Oil and Petrolatum by means of A.S.T.M. Union Colorimeter (D 155 – 39 T),
Test for Consistency of Lubricating Greases and Petrolatum (D 217 – 38 T),
Test for Sediment in Fuel Oil by Extraction (D 473 – 38 T),
Test for Ash Content of Petroleum Oils (D 482 – 38 T),
Specifications for Fuel Oils (D 396 – 39 T), and
Specifications for Gasoline (D 439 – 40 T).

TABLE IV.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. New Tentative Standards			
Test for Neutralization Number of Petroleum Products by Color-Indicator Titration	42	2	8
Test for Neutralization Number of Petroleum Products by Electrometric Titration	39	2	11
Test for Rust-Preventing Characteristics of Steam-Turbine Oils in Presence of Water	41	2	9
Conversion of Kinematic Viscosity to Saybolt Furol Viscosity	48	0	1
II. Revisions of Tentative Standards			
Test for Gum Stability of Gasoline (D 525 – 41 T)	43	1	8
Test for Knock Characteristics of Aviation Fuels (D 614 – 41 T)	35	1	13
Test for Knock Characteristics of Motor Fuels (D 357 – 41 T)	38	3	11
Test for Saponification Number (D 94 – 41 T)	46	1	5
Test for Kinematic Viscosity (D 445 – 39 T)	47	0	5
III. Revisions of Standards, Immediate Adoption			
Test for Gum Content of Gasoline (D 381 – 36)	45	0	7
Test for Flash Point by Means of the Pensky-Martens Closed Tester (D 93 – 40)	50	0	2
Test for Melting Point of Paraffin Wax (D 87 – 37)	43	0	9
Test for Vapor Pressure of Petroleum Products (Reid Method) (D 323 – 41)	43	0	9
IV. Adoption of Tentative Standards as Standard			
Test for Carbon Residue of Petroleum Products (Ramsbottom Carbon Residue) (D 524 – 41 T)	47	3	2
Test for Dropping Point of Lubricating Grease (D 566 – 40 T)	43	1	5
Test for Tetraethyl Lead in Gasoline (D 526 – 41 T)	42	1	9
V. Withdrawal of Tentative Standard			
Test for Neutralization Number of Petroleum Products and Lubricants (D 188 – 41 T)	43	4	5

It is recommended that these tentative standards be continued in the tentative status because further developments are in progress or in prospect.

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[19] 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 259.

[20] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 208.

[21] 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 313.

[22] 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 302.

VII. STANDARDS SIX YEARS WITHOUT REVISION

The following standards have been published for six years or longer without revision and are considered by the committee to be in accord with present practice and requiring no revision:

Test for Autogenous Ignition Temperatures of Petroleum Products (D 286 – 30),
Test for Burning Quality of Long-Time Burning Oil for Railway Use (D 219 – 36),
Test for Burning Quality of Mineral Seal Oil (D 239 – 30),
Test for Dilution of Crankcase Oils (D 322 – 35),
Test for Steam Emulsion of Lubricating Oils (D 157 – 36),
Test for Flash and Fire Points by Means of Open Cup (D 92 – 33),
Test for Flash Point by Means of the Tag Closed Tester (D 56 – 36),
Test for Melting Point of Petrolatum (D127 – 30),
Test for Detection of Free Sulfur and Corrosive Sulfur Compounds in Gasoline (D 130 – 30),
Sampling Petroleum and Petroleum Products (D 270 – 33), and
Abridged Volume Correction Table for Petroleum Oils (D 206 – 36).

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 73 voting members, with the results shown in Table IV.

The present officers of the committee were re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 73 voting members; 52 members returned their ballots, of whom 51 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

T. A. BOYD,
Chairman.

R. P. ANDERSON,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee D-2 presented to the Society through Committee E-10 on Standards proposed Emergency Alternate Provisions in the Standard Method of Test for Carbon Residue of Petroleum Products (Ramsbottom Carbon Residue) (D 524 – 42) which were accepted by Committee E-10 on July 29, 1942, and issued under the designation EA – D524. They were published in ASTM BULLETIN, No. 118, October, 1942, p. 55, and have been issued in the form of a pink sticker for attachment to the standard.

REPORT OF SUBCOMMITTEE VI ON COLOR

For the past two years, Subcommittee VI on Color has been studying the application of the photoelectric colorimeter to the measurement of color of lubricating oil. The uncertainty of the future supply of satisfactory Union Colorimeter glass color standards[1] added impetus to the work.

The Proposed Method of Test for Color of Lubricating Oil by Means of Photoelectric Colorimeter, appearing in Appendix II, is the result of the efforts of the subcommittee. It is described in general terms to permit the use of any photoelectric colorimeter which meets the requirements prescribed. The method specifies an instrument which may be standardized by means of six aqueous solutions; thus insuring reproducibility. Since only a meter scale is read, the results are independent of the color sense and judgment of the operator. The method provides a continuous scale for obtaining the color of all lubricating oils, the color of which can be determined by the A.S.T.M. Union Colorimeter method.[1]

The apparatus may be used to obtain additional information concerning the spectral distribution of the sample. Also, data obtainable by use of the apparatus may be employed to calculate such I.C.I. values as tristimulus values, trichromatic coefficients, etc.

Preliminary observations were conducted by the laboratory personnel of 13 companies. The comments were generally favorable, the criticisms being based primarily on constructional details.

The major cooperative work on the method took place at a meeting of the representatives of 6 companies. Three instruments and ten oil samples were employed, and 55 observations were made. With one exception, readings of the individual oils checked within plus or minus 0.5 scale unit. The representatives of the other 7 companies used their own samples in conducting their observations.

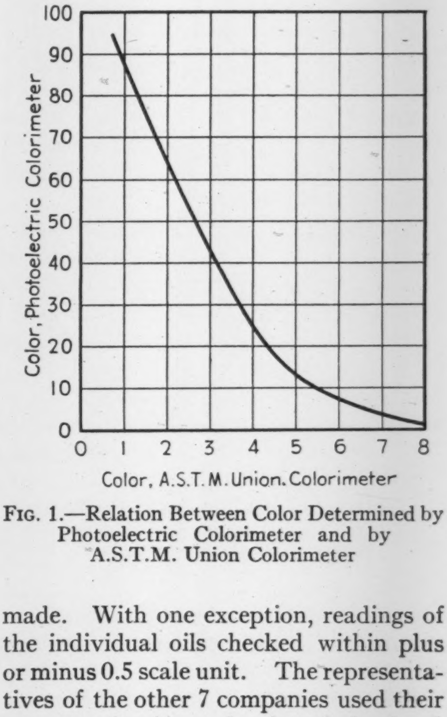

FIG. 1.—Relation Between Color Determined by Photoelectric Colorimeter and by A.S.T.M. Union Colorimeter

The relation between color determined by the photoelectric colorimeter and by the A.S.T.M. Union Colorimeter is shown in Fig. 1. This figure is based on the tristimulus values of the primary standard Union Colorimeter glasses measured by the National Bureau of Standards and on similar data obtained in the laboratories of Socony-Vacuum Oil Co. using a commercial instrument which complies with the requirements of the method. All of the readings obtained in the cooperative studies agree with this curve within a quarter unit of A.S.T.M. Union Color equivalent. Obviously, if the individual glasses in a Union Colorimeter do not conform closely to the primary standards, a greater deviation is possible.

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[1] Tentative Method of Test for Color of Lubricating Oil and Petrolatum by Means of A.S.T.M. Union Colorimeter (A.S.T.M. Designation: D 155), 1942 Book of A.S.T.M. Standards, Part III, p. 905

Respectfully submitted on behalf of the subcommittee,

H. M. HANCOCK,
Chairman.

REPORT OF TECHNICAL COMMITTEE C ON TURBINE OILS

Technical Committee C on Turbine Oils was organized early in 1940 in response to requests by turbine manufacturers and users. The following subjects were indicated as the most important:

1. *Design and Use.*—There are significant factors in the design and use of turbines which influence the life of the oil used to lubricate these installations.
2. *Rusting.*—While previous work in Committee D-2 has dealt with various characteristics of turbine oils, no attention has been given to rusting behavior, which had become an important question in the past few years.
3. *Oxidation Stability.*—The basic question of oxidation stability is one with respect to which there has been no general agreement as to method of appraisal.

Accordingly, the program of Technical Committee C was organized to treat these three subjects, and the work to date of the corresponding sections is summarized in this report.

Section I on Oil Systems of Turbines (F. E. Rosenstiehl, chairman) has functioned by serving as a medium of interchange of information and ideas between representatives of turbine manufacturers, oil producers, and turbine users. A number of meetings have been held, at which various aspects of the problems involved have been discussed. Informal papers have been presented on oil-purification systems and on the cause and prevention of foaming of turbine oils. Materials of construction have been dealt with from the standpoints of the influence of various metals, notably copper, in accelerating the oxidation of oils, and of the use of protective coatings for tanks and other parts of the turbine system. Some information on the design of tanks, sumps, and means of venting has also been made available. Perhaps the most comprehensive work of the section has been the development of a questionnaire which was sent to turbine users by the Power Station Chemistry Subcommittee of the Prime Movers Committee of the Edison Electrical Institute. Information obtained in this questionnaire on temperatures and other factors in oil usage is now being assembled and correlated.

Section II on Rusting (W. P. Kuebler, chairman) has studied rusting tests based on general recognition that the rust-preventive characteristic of turbine oils is an important quality affecting performance. The first set of cooperative tests involved the use of non-standardized tests, such as were in use at the various laboratories, on a series of new turbine oils. These tests included (*a*) several variations of a procedure developed in the laboratory of the Westinghouse Electric and Manufacturing Co. involving contact of a strip of steel with a stirred mixture of the oil with a small amount of water,[1] and (*b*) a procedure in which the strip of steel is dipped alternately in the oil and water. The general conclusion from these tests was that the different procedures showed the same general trends, and the stirring type of test (*a*) was selected for further study. The results showed that the rusting was more severe as the temperature of test was increased, and it was decided that temperatures approximating service conditions were preferable.

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[1] C. Dantsizen, "Lubrication of General Electric Steam Turbines," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 63, No. 6, August, 1941.

With a preliminary standardization of variables, a second set of cooperative tests was run on oils on which a definite rating as to rusting tendencies was available, on the basis of plant performance. In these tests, the oils were rated in the same order by each laboratory and the rating agreed with the service data, but the absolute degree of rusting for a given oil varied considerably. It was felt that these variations were due to some differences in technique, particularly in the preparation of the test strips. Three modifications of the method of polishing the strip were under consideration, and in the third series of tests the simplest and most practical polishing method gave the most consistent data. In this series, three oils of known service behavior were tested and Table I shows the results submitted by ten laboratories. It will be noted that all of the oils were clearly classified in line with their service behavior; the critical sample, No. 14, permitted a slight degree of rusting which varied but little in the separate laboratories. The technique used in this set of tests is recommended for publication as a Tentative Method of Test for Rust-Preventing Characteristics of Steam Turbine Oil in the Presence of Water.[2]

TABLE I.—RESULTS OF COOPERATIVE RUSTING TESTS BY PROPOSED PROCEDURE.

Laboratory	Ratings of Oil Samples[a]		
	No. 13	No. 14	No. 15
A	severe	very light	none
B	severe	few spots	none
C	severe	trace	none
D 1	severe	few spots	none
2	severe	few spots	none
E	severe	light	none
F	severe	few spots	none
G	severe	very light	none
H	severe	slight	none
I	severe	slight	none
J	severe	slight	none

[a] Service rating of oils tested:
No. 13—A new oil, with which marked corrosion is experienced.
No. 14—Borderline oil; will permit very little corrosion.
No. 15—Definitely inhibits corrosion even in concentration of 10 per cent by volume in new, noninhibiting oil.

Section III on Oxidation of Turbine Oils (C. Dantsizen, chairman) has carried out one set of cooperative tests using a published method[3] which involves long-continued oxidation of oil at 95 C. in the presence of water and coils of iron and copper wire. Deterioration of the oils was determined by rise in neutralization number and also in diminution of interfacial tension toward water. The service life of the oils used in this set of tests is not known definitely. In view of this lack, as well as the need for standardizing and determining the effect of various factors in the observation test, no recommendations are being made at this time. The work of this section will be continued and an effort will be made to make available oils of known service behavior.

Respectfully submitted on behalf of the technical committee,

F. C. LINN,
Chairman.

T. H. ROGERS,
Secretary.

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[2] This method was accepted as tentative by the Society and appears in the 1942 Book of A.S.T.M. Standards, Part III, p. 969.

[3] G. H. von Fuchs, N. B. Wilson, and K. R. Edlund, "The Evaluation and Performance of Turbine Oils," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 13, No. 5, May, 1941, p. 306.

APPENDIX I

PROPOSED METHOD OF TEST FOR OIL CONTENT OF PARAFFIN WAX[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia, Pa.

Scope

1. This method of test is intended for the determination of the percentage of oil in paraffin wax having a melting point of 105 F.[2] or higher, and containing not more than 10 per cent by weight of oil.

Nature of Test

2. The method involves dissolving the wax in methylethylketone, cooling to minus 25 F. to precipitate the wax, filtering out the methylethylketone containing the dissolved oil, and determining the oil content of this filtrate by evaporating the methylethylketone and weighing the residue.

Apparatus

3. The apparatus shall consist of the following:

(*a*) *Cooling Vessel.*—A cooling vessel, substantially V-shaped, with approximate dimensions as follows:

Depth	10 in.
Length	4⅛ in.
Width at top of V	2 in.
Radius of curvature at bottom of V	$\frac{9}{16}$ in.

A suitable cooling vessel is shown in Fig. 1.

(*b*) *Filter Leaf.*—A filter leaf of any convenient shape, approximately 1 in. in thickness and not more than 4 in. in height. A suitable filter leaf is shown in Fig. 2. The filter medium for the leaf shall be qualitative filter paper,[3] supported by a perforated metal plate. Alternatively a leaf covered tightly with filter canvas (No. 8 cotton duck), previously washed free of soluble matter with methylethylketone, may be used. A metal tube at least 12 in. in length, ¼ in. in outside diameter, and ⅛ in. in inside diameter shall be provided, running through, and sealed to, the frame, and extending to within ⅛ in. of the bottom of the leaf, inside the paper or cloth.

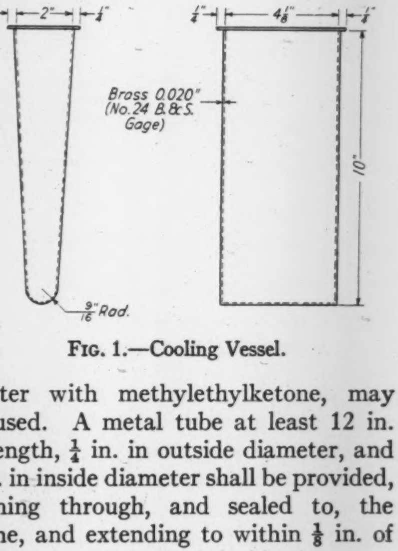

FIG. 1.—Cooling Vessel.

(*c*) *Precooling Chamber.*—A precooling chamber 10 in. in depth with other dimensions such as to provide a loose fit for either one or two filter leaves.

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. Published as information, June, 1942.

[2] Determined in accordance with the Standard Method of Test for Melting Point of Paraffin Wax (A.S.T.M. Designation: D 87) of the American Society for Testing Materials, 1942 Book of A.S.T.M. Standards, Part III, p. 189.

[3] Eaton—Dikeman No. 615, or equivalent.

A suitable precooling chamber for a single filter leaf is shown in Fig. 3.

(*d*) *Cooling Bath.*—A cooling bath of a size to accommodate any desired number of cooling vessels and precooling chambers, and designed to maintain a temperature of minus 35 ± 5 F. The bath may be filled with a suitable medium such as methylethylketone and may be cooled by circulating a refrigerant through coils, or by using solid carbon dioxide.

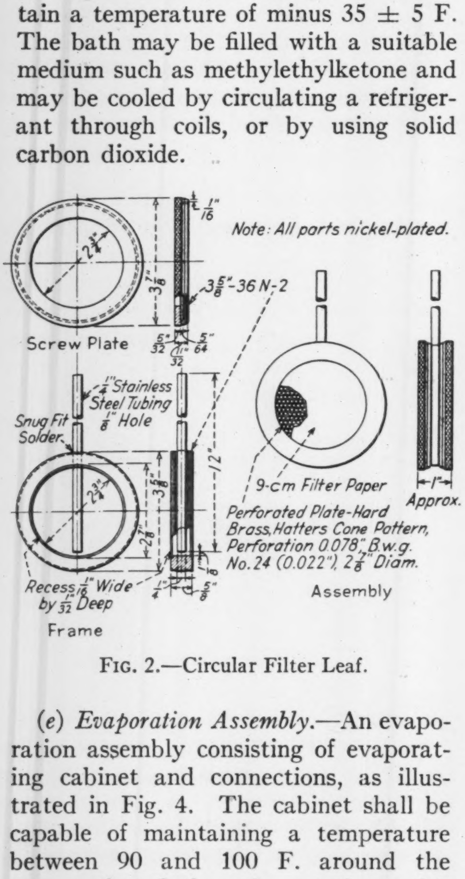
1"/16
Note: All parts nickel-plated.
2 3/4"
3 7/8"
3 5/8"-36 N-2
Screw Plate
5"/32
1"/32
5"/64
1/4" Stainless Steel Tubing
1/8" Hole
Snug Fit Solder
2 3/4"
3 5/8"
2 7/8"
12"
1/8"
9-cm Filter Paper
1"
Approx.
Perforated Plate-Hard Brass, Hatters Cone Pattern, Perforation 0.078", B.w.g. No. 24 (0.022"), 2 7/8" Diam.
Recess 1/16" Wide by 1/32" Deep
1/4"
5/8"
Frame
Assembly

FIG. 2.—Circular Filter Leaf.

(*e*) *Evaporation Assembly.*—An evaporation assembly consisting of evaporating cabinet and connections, as illustrated in Fig. 4. The cabinet shall be capable of maintaining a temperature between 90 and 100 F. around the evaporation flasks. As an alternative to the evaporating cabinet a water bath, equipped with heating and stirring devices capable of maintaining a temperature of 95 ± 2 F., may be used and the air stream preheated to this temperature by passage through a metal coil immersed in the water bath (see Fig. 5). Jets shall be provided for delivering a stream of clean, dry air vertically downward into the evaporation flasks. Th inside diameter of each jet shall be 3 ± 0.2 mm., and the tip of each jet shall b 25 ± 5 mm. above the surface of th liquid at the start of evaporation. Ai shall be supplied at the rate of 2 to liters per minute. The air shall b purified by passage through a com mercial air filter and a 30-cm. column of 30- to 60-mesh fuller's earth.[4] The cleanliness of the air shall be checked periodically by evaporating 100 ml. of methylethylketone by the procedure specified in Section 5 (*f*). The residue shall not exceed 2 mg.

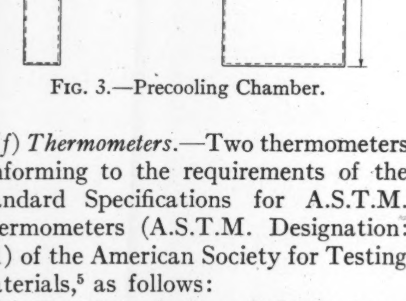
1/4"
1/8"
1/4"
1/4"
4 1/8"
1/4"
Brass 0.020" (No. 24 B.&S. Gage)
10"

FIG. 3.—Precooling Chamber.

(*f*) *Thermometers.*—Two thermometers conforming to the requirements of the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1) of the American Society for Testing Materials,[5] as follows:

(*1*) For the solution of the wax, an A.S.T.M. Partial Immersion Thermometer E 1 (1 F – 39), having a range of 0 to 300 F.

(*2*) For the cooling bath and cooling vessel, an A.S.T.M. Low Cloud and Pour Thermometer E 1 (6 F – 39), having a range of –70 to +70 F.

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[4] The life of the fuller's earth drying column can be lengthened by placing a gas washing bottle containing sulfuric acid (95 per cent) before the drying column.

[5] 1942 Book of A.S.T.M. Standards, Part II, p. 974; Part III, p. 818.

Solvent

4. *Methylethylketone.*—The technical grade is satisfactory if its refractive index at 68 F. (20 C.) is 1.378 ± 0.002 and if the residue remaining after the evaporation of 100 ml. by the procedure specified in Section 5 (*f*) does not exceed 2 mg.

Procedure

5. (*a*) Melt a representative portion (Note 1) of the sample of wax in a suitable container, using a water bath or oven maintained at 160 to 180 F. Pour 24 to 26 g. of the sample, as soon as possible after melting, into a clean, dry 500-ml. Erlenmeyer flask weighed to the nearest 0.2 g. Weigh the flask and sample to the nearest 0.2 g., and add 375 ± 5 ml. of methylethylketone. Attach a water-cooled condenser to the flask by a ground-glass joint or cork covered with metal foil, and heat the flask and contents until the sample has dissolved, taking care to prevent surging of the liquid in the condenser.

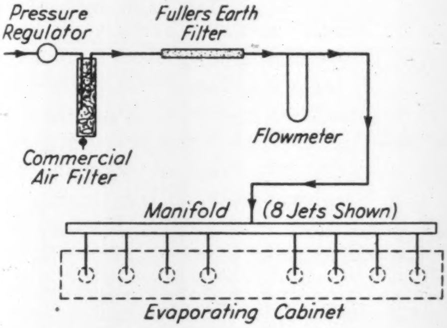

FIG. 4.—Evaporation Assembly.

NOTE 1.—If the sample of wax is 2 lb. or less, obtain a representative portion by melting the entire sample and stirring thoroughly. For samples over 2 lb., exercise especial care to insure obtaining a truly representative portion, bearing in mind that the oil may not be distributed uniformly throughout the sample, and that mechanical operations may express some of the oil.

(*b*) Discontinue heating and, after boiling ceases, disconnect the condenser. Weigh rapidly the flask and contents to the nearest 0.5 g. Then close the flask with a stopper carrying the partial immersion thermometer.

(*c*) When the solution has cooled to about 130 F., but before any wax precipitates, pour the solution into the clean, dry, cooling vessel (Fig. 1) contained in the cooling bath at minus 35 ± 5 F. and stir frequently. When the temperature of the mixture in the cooling vessel reaches minus 20 F., stir constantly until the temperature reaches minus 25 ± 0.5 F.

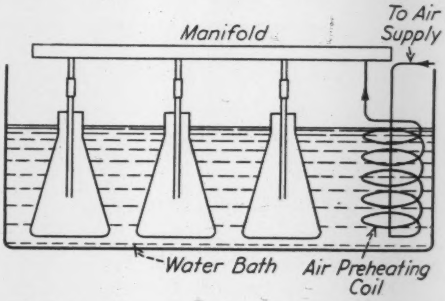

FIG. 5.—Alternative Evaporation Assembly.

(*d*) Immerse the clean, dry filter leaf or leaves, previously cooled to minus 35 F. in the precooling chamber (Fig. 3), in the mixture and connect to a 250-ml. Erlenmeyer flask weighed with a tare to the nearest 1 mg., and to a suction line on which a partial vacuum of at least 10 in. of mercury is maintained (See Fig. 6). Collect about 100 ml. of filtrate in the Erlenmeyer flask.

(*e*) Disconnect the Erlenmeyer flask, rinse the outside of the flask with acetone, wipe dry with a clean cloth, and weigh the flask and contents to the nearest 0.2 g.

(*f*) Place the flask in the evaporation assembly maintained at 90 to 100 F. with the air jet inside the neck of the flask and the tip 25 ± 5 mm. above the surface of the liquid (Note 2). After 2 hr. of evaporation, allow the flask to cool to room temperature and weigh the Erlenmeyer flask and contents with a tare to the nearest 1 mg. Continue evaporation as before, weighing at hourly intervals until the loss between successive weighings is not over 2 mg.

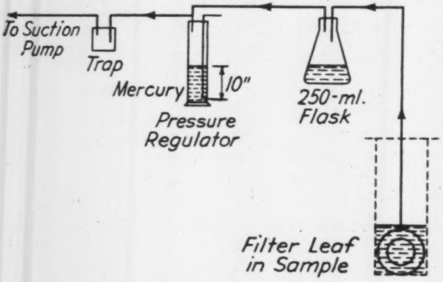

FIG. 6.—Filtration Assembly.

NOTE 2.—If a water bath is used as an alternative to the evaporating cabinet as permitted under Section 3 (*e*), modify the evaporation procedure as follows: Place the flask in the water bath maintained at 95 ± 2 F. with the air jet inside the neck of the flask and the tip 25 ± 5 mm. above the surface of the liquid. After 2 hr. of evaporation, remove the flask from the water bath, rinse the outside of the flask with acetone to remove water, place on paper towel or other blotter to dry for 1 min., and cool for 15 min. in a desiccator containing calcium chloride. Do not wipe the flask with a towel as this may create static, which is objectionable during weighing. Weigh flask and contents with a tare to the nearest 1 mg. Continue evaporation as before, weighing at intervals of 30 min., until the loss between successive weighings is not over 2 mg.

Calculation

6. Calculate the percentage of oil i the wax as follows:[6]

Oil in wax, per cent by weight =

$$\left(100 \times \frac{A}{B} \times \frac{C}{D}\right) - 0.1$$

where:

A = weight in grams of oil residue,
B = weight in grams of wax sampl
C = weight in grams of solvent—obtained by subtracting weight c 500-ml. Erlenmeyer flask plu wax sample (Section 5 (*a*) from weight of flask and con tents (Section 5 (*b*)),
D = weight in grams of filtrate, and
0.15 = an average factor correcting fo the solubility of wax in th solvent at —25 F.

If the result is negative, it shall b reported as zero.

Reproducibility of Results

7. With care and proper attention t details, duplicate results, expressed a percentage of oil in the wax, should no differ from the average by more tha the amounts shown in the followin tabulation:

Oil in the Wax, per cent	Numerical Deviation of Individual Results from Average	
	Results by One Operator	Results by Different Operators
Less than 1..........	0.1	0.2
1 and over...........	0.2	0.4

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[6] This formula does not provide for deduction of th weight of the oil residue from the weight of the filtrate and hence is only approximate. However, the results obtained are within the limits of reproducibility of th method, and the formula is simpler to use.

APPENDIX II

PROPOSED METHOD OF TEST FOR COLOR OF LUBRICATING OIL BY MEANS OF PHOTOELECTRIC COLORIMETER[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia, Pa.

Scope

1. This method of test is intended for the determination of the color of lubricating oil by means of a photoelectric colorimeter.

Nature of Test

2. The color of the oil is measured with a colorimeter consisting essentially of a stable source of illumination, a standard sample holder, a light filter, and a photoelectric cell. The measured color is fundamental in nature because of its direct relation to the I.C.I. "Y" tristimulus value or brilliance of the sample. Photoelectric color plotted against the "Y" tristimulus value yields a curve which is a straight line passing through the origin and having a slope of 0.90. This slope results from the use of distilled water as a standard, and from the fact that the brilliance of distilled water against air or vacuum is approximately 90 per cent.

Apparatus

3. The component parts of the photoelectric colorimeter shall conform to the following requirements:

(*a*) *Source of Illumination.*—The source of illumination shall be an incandescent light bulb supplied in such a manner that the intensity of light output is independent of fluctuation of plus or minus 15 per cent in the supply voltage, and arranged so that the focus is fixed and reproducible.

(*b*) *Optical System.*—The light from the lamp shall be filtered through a heat absorber, then passed through a system of lenses and diaphragms that will converge the beam to a spot substantially at the center of the sample container, after which the beam shall diverge.

(*c*) *Sample Container.*—The sample container shall be a round tube with closed bottom, made of Pyrex glass, or its equivalent. The sample container shall have an outside diameter of approximately 20 mm. and a length of about 140 mm. Each sample tube of a set shall be interchangeable with any other tube of the set within a tolerance of 0.1 photoelectric color units. The container should be so supported that it is reproducibly positioned upon random insertion.

(*d*) *Filters.*—The light filters shall be made of glass, or combinations of glass, in which the coloring material shall be uniformly dispersed. The filters shall retain their colors indefinitely. Three filters, designated as red, violet, and north sky filters, shall be provided.

(*e*) *Electrical Circuit.*—The electrical circuit shall be such that no fatigue shall be observable, and shall include a control capable of coarse and fine adjustment for setting the pointer at 100.

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. Published as information, June, 1942.

The experimental work conducted in the development of this method was performed with Hellige-Diller colorimeters.

(*f*) *Photoelectric Cell.*—The photoelectric cell shall be of the self-generating, barrier-layer type.

(*g*) *Meter.*—The meter shall consist of an instrument to measure the output of the photoelectric cell, with a linear scale indicating zero in the dark and 100 with distilled water in the sample container. The meter shall be able to withstand an overload of 1000 per cent, and its pointer shall be at all times under damping control.

Standardization of Apparatus

4. (*a*) The apparatus shall be standardized as a complete unit, so that standard solutions placed in the sample container will yield certain fixed scale deflections with each of the three filters. The following solutions prepared with c.p. chemicals shall be used for this standardization:

Solution 1, Sulfuric Acid.—Stir 300 g. of H_2SO_4 (sp.gr. 1.84) in distilled water to make 1 liter of solution.

Solution 2, Ammonium Hydroxide.—Stir 250 ml. of NH_4OH (sp.gr. 0.90) in distilled water to make 1 liter of solution.

Solution 3, Alkaline Borax Solution.—Dissolve 10 g. of borax ($Na_2B_4O_7 \cdot 10H_2O$) and 30 g. of KOH in distilled water and dilute to 1 liter.

Solution 4a, Ammoniacal Nickel Sulfate Solution.—Dissolve approximately 2.5 g. of $NiSO_4 \cdot 6H_2O$ in solution 2 and dilute to approximately 100 ml. with solution 2. Determine accurately by analysis the nickel content of the solution; dilute with solution 2 to give 0.500 g. of nickel per 100.0 ml. of solution. Restandardize, and readjust if necessary.

Solution 4b, Diluted Ammoniacal Nickel Sulfate Solution.—Dilute 25.0 ml. of solution 4*a* to 100.0 ml. with solution 2.

Solution 5a, Acid Potassium Dichromate Solution.—Dissolve exactly 10.000 g. of dry, c.p. $K_2Cr_2O_7$ in solution 1, and dilute to exactly 100.0 ml. with solution 1.

Solution 5b, Diluted Acid Potassium Dichromate Solution.—Dilute 10.0 ml. of solution 5*a* to 100.0 ml. with solution 1.

Solution 6a, Potassium Dichromate - Alkaline Borax Solution.—Dissolve 0.0200 g. of pure, dry $K_2Cr_2O_7$ in solution 3, and dilute to 100.0 ml. with solution 3.

Solution 6b, Diluted Potassium Dichromate-Alkaline Borax Solution.—Dilute 10.0 ml. of solution 6*a* to 100.0 ml. with solution 3.

NOTE 1.—If these solutions are to be kept, they shall be stored in glass-stoppered, alkali-resistant glass bottles. Even with this precaution, the solutions may develop a very fine precipitate. The photoelectric color reading is affected by turbidity too slight to be visible to the eye. All of these solutions, therefore, shall be filtered through clean, dry asbestos before use. This filtration does not noticeably affect the content of the colored ion.

(*b*) To standardize the apparatus, measure the color of the standard solutions in exactly the same manner as in the measurement of the color of a sample (Section 5). Under these conditions, the percentage transmittances obtained on the standard solutions, with the appropriate filters, shall agree with the values shown in Table I within

TABLE I.—TRANSMITTANCE WITH STANDARD SOLUTIONS.

Standard Solution	Transmittance, per cent		
	North Sky Filter	Red Filter	Violet Filter
4*a*.......	48	44	79
4*b*.......	..	80	..
5*a*.......	32	99	..
5*b*.......	49	..	1
6*a*.......	94	100	24
6*b*......	..	..	70

plus or minus 1 unit. These readings shall be obtainable at any desired time, regardless of the age of the instrument.

Procedure

5. (*a*) Test the sample at room temperature, unless heat is necessary to remove cloud or to melt the sample. To remove cloud, heat the sample to a temperature not more than 10 F. (5.6 C.) above the cloud point. When it is necessary to melt the sample, heat it to a temperature 20 to 30 F. (11 to 17 C.) above the melting point.

(*b*) Using the North sky filter, set the colorimeter to read 100 per cent transmittance with distilled water in the cell. Measure the percentage transmittance of the sample, reading to the nearest 0.5 unit if above 5, and to the nearest 0.1 unit (estimated) if between 0.2 and 5. Recheck the setting on distilled water and repeat the measurement if this setting has varied by more than 0.5 unit. If

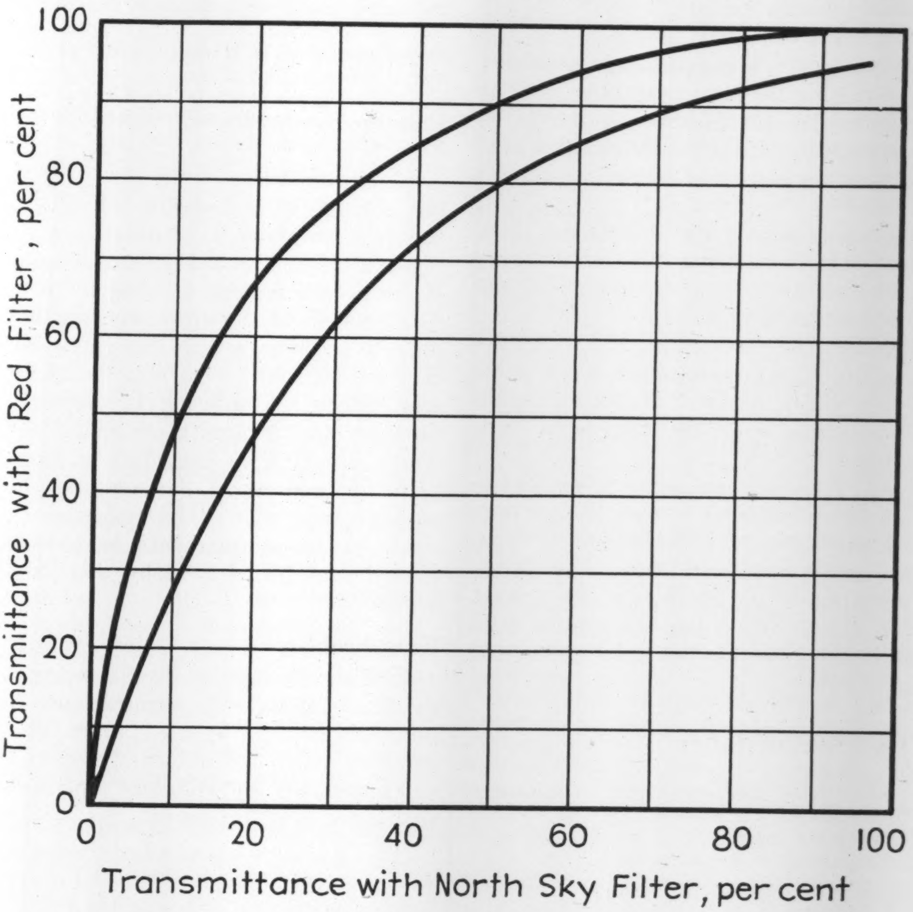

FIG. 1.—Transmittance with Red Filter *versus* Transmittance with North Sky Filter.

the transmittance of the sample with the North sky filter is between 0.2 and 96 per cent, proceed in accordance with Paragraph (*c*); if the transmittance is above 96 per cent, proceed in accordance with Paragraph (*d*); if the transmittance is below 0.2 per cent, proceed in accordance with Paragraph (*e*).

(*c*) *Transmittance Between 0.2 and 96 per cent.*—The percentage transmittance using the North sky filter shall be reported as the photoelectric color of the sample. If further information concerning the sample is desired, repeat the procedure described in Paragraph (*b*), including setting the colorimeter with distilled water, using the red filter in place of the North sky filter. If the transmittance with the red filter plotted against the transmittance with the North sky filter does not give a point lying within the band shown on Fig. 1, the sample shall be considered "abnormal."

(*d*) *Transmittance Above 96 per cent.*—Set the colorimeter to read 100 per cent transmittance with distilled water using the violet filter, and measure the transmittance of the sample to the nearest 0.5 unit. Report to the nearest 0.05 unit the photoelectric color calculated as follows:

$$\text{Photoelectric color} = 90.0 + \frac{\text{percentage violet transmittance}}{10}$$

If the transmittance, using the North sky filter, obtained as described in Paragraph (*b*) differs by more than 0.5 unit from the photoelectric color as calculated the sample shall be considered "abnormal."

NOTE 1.—The beam of light passing through the sample may cause a change in the color of unstable products. With such samples, the measurement shall be made as rapidly as possible to minimize this effect.

NOTE 2.—If the sample is turbid, or contains suspended matter, filter it through clean, dry paper before measurement.

(*e*) *Transmittance Below 0.2.*—Dilute the sample with kerosine of photoelectric color above 98 to obtain a solution containing 15 ± 0.1 per cent sample. If the sample requires heating to 20 to 30 F. (11 to 17 C.) above the melting point in accordance with Paragraph (*a*), heat the kerosine to the same temperature before the solution is prepared. Measure the transmittance using the North sky filter as described in Paragraph (*b*) and report the result as the photoelectric color. The photoelectric colors of all samples that have been diluted shall be followed by the designation "dil."

Reproducibility

6. With care, and proper attention to details, duplicate determinations of photoelectric color by one operator shall agree to within 0.5 unit, and duplicate determinations by different operators shall agree to within 1 unit.

APPENDIX III

PROPOSED METHOD OF TEST FOR POTENTIAL GUM IN AVIATION GASOLINE[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia, Pa.

1. *Scope*.—This method describes a procedure for an accelerated oxidation stability test for aviation gasoline by means of which the quantity of residue (gum plus lead deposit) is measured after the gasoline has been oxidized for a specified time under prescribed conditions. Provision is made for a correction in the case of aviation gasoline containing tetraethyl lead.

2. *Apparatus:* (*a*) *Bomb*.—The bomb shall be made of a stainless steel (Note 1) with the inside dimensions of the portion that encloses the reacting gasoline-oxygen mixture conforming substantially to those shown in Fig. 1. The interior surface of the bomb and lid shall be given a high polish to facilitate cleaning and to prevent corrosion. Other structural details such as the method of closure, gasket material, and outside dimensions are optional, provided the following limitations are observed:

(*1*) The bomb shall be constructed to withstand a working pressure of 180 psi. at 212 F. (100 C.), with an ultimate strength at least equal to that of a bomb constructed of 18 per cent chromium, 8 per cent nickel alloy steel (Note 1) and having dimensions as shown in Fig. 1.

(*2*) The closure (Note 2) shall be capable of making a seal that will not leak when the bomb is filled with oxygen to 100 psi. at 60 to 80 F. (15.6 to 26.7 C.), and plunged into a bath at 212 F. (100 C.). Any suitable gasket material may be used that will not react with oxygen (Note 3).

(*3*) The weight of the bomb assembled as shown in Fig. 1, but without the oxygen valve and glass liner, shall be not less than 5.0 lb. nor more than 8.0 lb. This weight shall be distributed about the sample container in a manner equivalent to that shown in Fig. 1.

Note 1.—A suitable material is an 18 per cent chromium, 8 per cent nickel alloy steel conforming to grade S, type 304 of the Standard Specifications for Corrosion-Resisting Chromium and Chromium-Nickel Steel Sheet, Strip, and Plate for Fusion-Welded Unfired Pressure Vessels (A.S.T.M. Designation: A 240) of the American Society for Testing Materials;[2] or S.A.E. Nos. 30905 or 30915.[3]

Note 2.—It is preferable that the closure ring be constructed from an alloy different from that used for the bomb body, particularly if the bomb metal is that specified in Note 1 and if the mating threads of the two parts are to move with respect to each other when a tightening load is applied.

Note 3.—Reactivity with oxygen shall be determined by the following test: The gasket under test shall be placed in the bomb in the absence of gasoline and a gasket of similar composition used in the attachment of the cover. The bomb shall be filled with oxygen to a pressure of 100 psi. and immersed in a bath at about 212 F. (100 C.). If the pressure does not drop more than 2 lb. from the maximum in a 24-hr. period, with the bath temperature differing by not more than 2.0 F. from that when the pressure was at its maximum, the gasket shall be considered nonreactive in the presence of oxygen.

(*b*) *Glass Liner*.—A glass liner for the bomb shall be provided, the shape and dimensions of which shall conform to those shown in Fig. 1. The glass liner shall fit snugly inside the bomb.

(*c*) *Bomb Accessories*.—The bomb lid shall be provided with a stem and stem filler rod, both constructed of stainless steel and having dimensions substantially as shown in Fig. 1. The inside of the stem and the outside of the filler rod shall be given a high polish to facilitate cleaning. Means shall be provided for connecting to the bomb stem an indicating or recording pressure gage (Paragraph (*d*)) and a tightly closing needle valve (Note 4) through which oxygen may be admitted. The connection to the source of oxygen may be made as indicated in Fig. 1, or it may be made through a tee or cross connection on the top of the bomb stem. A circular metal plate of a diameter sufficient to serve as a lid for closing the bath when the bomb is in place shall be fitted to the stem in any convenient manner but in the position shown in Fig. 1. A suitable holder for the bomb and an adequate wrench for obtaining a tight closure shall be provided.

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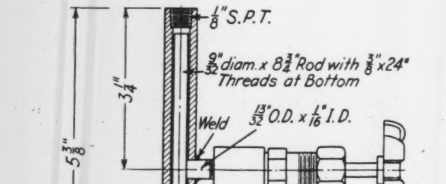
[1] **This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. Published as information, June, 1941; modified June, 1942.**

[2] 1942 Book of A.S.T.M. Standards, Part I, p. 467.

[3] **1942 S.A.E. Handbook, p. 308.**



$\frac{1}{8}$" S.P.T.
$\frac{9}{32}$" diam. x $8\frac{3}{4}$" Rod with $\frac{3}{8}$" x 24" Threads at Bottom
Weld
$\frac{13}{32}$" O.D. x $\frac{1}{16}$" I.D.
$3\frac{1}{4}$"
$5\frac{3}{8}$"
$\frac{3}{16}$" Approx.
$\frac{1}{8}$" S.P.T. Angle Valve
$3\frac{1}{2}$"
$\frac{1}{8}$"
Weld
$\frac{5}{8}$"
$\frac{5}{16}$"
3"
$\frac{3}{8}$" x 24 Threads $\frac{3}{8}$" Long
Flat on Thread of Rod $\frac{1}{16}$" Deep x $\frac{9}{16}$" Long
$2\frac{1}{2}$"
$2\frac{1}{4}$"
2"
Weld
$\frac{3}{16}$" x $\frac{1}{8}$" Gasket
Octagon $2\frac{7}{8}$" Across Flats
$\frac{3}{8}$"
$\frac{5}{8}$"
1"
$1\frac{5}{8}$"
$\frac{1}{4}$"
$\frac{7}{8}$"
$3\frac{1}{8}$"
$2\frac{11}{16}$" x 9 Acme Threads per Inch
$\frac{3}{4}$"
Glass Liner
Octagon $2\frac{3}{8}$" Across Flats
$4\frac{7}{8}$"
$4\frac{3}{8}$" $^{+}_{-}\frac{1}{16}$"
2"
0.060" to 0.080"
3"
1" rad.
$1\frac{1}{4}$"
$2\frac{3}{8}$"

FIG. 1.—Bomb for Accelerated Oxidation.

NOTE 4.—For convenience in handling, it is suggested that a tire valve and stem be attached to a three-way pressure cock which in turn is attached to the needle valve and that the oxygen be introduced through a suitable high-pressure air hose and fitting. It is considered safer practice to release the gas in the bomb through the third unobstructed port of the three-way cock rather than through the small orifice of the tire valve. The needle valve must not be eliminated, as it is necessary to insure against leaks at this point during the test.

(*d*) *Pressure Gage.*—A pressure gage with a maximum reading of 200 psi. shall be used. It may be of either the indicating or the recording type. The scale for the range 100 to 180 psi. shall average at least 1 in. per 50 psi. pressure differential measured along the arc of the scale. Intervals of division shall be 5 psi. or less. The accuracy shall be 1 per cent of the total scale interval. The gage may be connected to the bomb stem directly or by a flexible metal tubing, but the total volume of the stem with filler rod in place and all connections shall be not more than 15 ml.

(*e*) *Oxidation Bath.*—The oxidation bath shall be a water bath having a capacity of not less than 4.5 gal. per bomb, and of such dimensions that the maintained depth of liquid is not less than 11.5 in. The bath shall be equipped with a condenser and a heating unit for keeping the water boiling continuously. It also shall have a thermometer well such that the 206 F. (96.7 C.) point of the thermometer (Paragraph (*f*)) shall be of the same level as the upper surface of the bath cover. A round opening of such a diameter as to accommodate the bomb and to fit the plate fastened to the bomb stem shall be provided. The top of the bomb lid shall be submerged at least 2 in. below the surface of the bath liquid.

(*f*) *Thermometer.*—The thermometer shall be an A.S.T.M. Saybolt Viscosity Thermometer, graduated in either Fahrenheit or Centigrade degrees as specified, having a range of 204 to 218 F. or 95 to 103 C. and conforming to the requirements for this thermometer as prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1) of the American Society for Testing Materials.[4]

(*g*) *Evaporation Bath.*—The evaporation bath shall consist of a closed vessel provided with a reflux condenser, a well into which the beaker fits, a coil for preheating the air, and a conical adapter to deliver the air into the center of the beaker. A suitable form of apparatus is shown in Fig. 2. A multiple bath having similar provisions is also satisfactory. The bath shall be filled to within 1 in. of the top with a stable liquid having a boiling point within a temperature range from 320 to 330 F. (160 to 165.8 C.). Ethylene glycol containing approximately 3 per cent water is suitable for the purpose. The bath shall be insulated with asbestos. An electric hot plate capable of keeping the liquid boiling actively is the preferred heating means. In the absence of a satisfactory hot plate, the bath may be heated by a gas burner, in which case the apparatus shall be placed in a hood with a good draft.

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[4] 1942 Book of A.S.T.M. Standards, Part II, p. 974; Part III, p. 818.

(*h*) *Catalyst.*—The catalyst shall be clean, sandblasted steel (Note 5), in the form of tubing or strips twisted into spirals, not exceeding $\frac{1}{32}$ in. in thickness and having a total surface of about 17.5 sq. in. Six tubes $\frac{1}{4}$ in. in outside diameter, $\frac{1}{32}$ in. in wall thickness, and 2 in. in length have sufficient surface and conveniently fit in the apparatus.

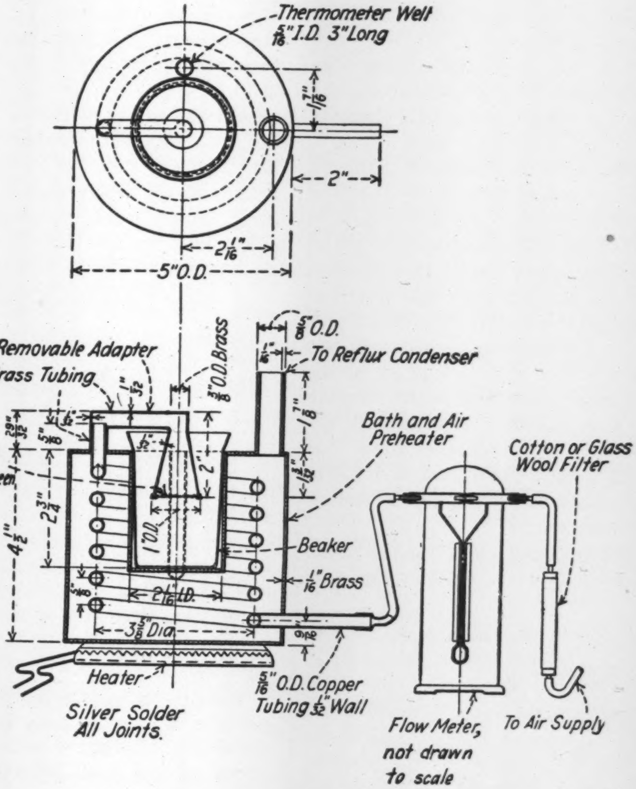
Thermometer Well
5/16" I.D. 3" Long
1 7/16"
2"
2 1/16"
5" O.D.
5/8" O.D.
1/4"
To Reflux Condenser
Removable Adapter
3/8" O.D. Brass Tubing
1 1/32"
3/8" O.D. Brass
1 7/8"
Bath and Air Preheater
Cotton or Glass Wool Filter
30-Mesh Copper Screen
29/32"
5/16"
1/2"
2"
2 3/4"
4 1/2"
1" O.D.
Beaker
1/16" Brass
2 1/16" I.D.
3 5/8" Dia.
Heater
5/16" O.D. Copper Tubing 1/32" Wall
Silver Solder All Joints.
Flow Meter, not drawn to scale
To Air Supply

FIG. 2.—Apparatus for Determining Gum Content of Oxidized Gasoline.

NOTE 5.—A suitable material is carbon steel conforming in chemical composition to grade 3 or 5 of the Standard Specifications for Commercial Quality Hot-Rolled Bar Steels (A.S.T.M. Designation: A 107) of the American Society for Testing Materials;[5] or S.A.E. No. 1020.[6]

(*i*) *Beakers.*—The beakers shall be of the Berzelius type, made of heat-resistant glass, without lip, and of 100-ml. nominal capacity.

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[5] 1942 Book of A.S.T.M. Standards, Part I, p. 117.

[6] 1942 S.A.E. Handbook, p. 304.

(*j*) *Gooch Crucible.*—A sintered-glass Gooch crucible, fine porosity, of 25-ml. nominal capacity shall be used.

(*k*) *Air Supply.*—Low-pressure air shall be passed through a cotton or glass-wool filter and delivered to the preheater inlet of the bath. A flowmeter or other air-measuring device shall be provided as shown in Fig. 2. The flowmeter shall be calibrated when attached to the bath with the bath liquid at room temperature.

3. *Procedure.*—(*a*) Carefully clean the bomb and its accessories before starting the test. Wash the glass liner with a suitable gum solvent (Note 6) and allow to dry. Soak the liner for a minimum of 12 hr. in chromic acid solution, rinse at least three times with distilled water, and finally dry by heating in an oven. The glass liner should be handled only with clean metal forceps after cleaning. Drain from the bomb any gasoline present and wipe the inside of the bomb and cap, first with a clean cloth moistened with the gum solvent and then with a clean dry cloth. Remove the gum or gasoline in the annular space with gum solvent. Occasionally remove the filler rod from the stem and carefully clean both stem and rod. The annular space shall be thoroughly dry before each test is started.

NOTE 6.—Either acetone or a blend of benzol and ethyl alcohol (1:1) is a satisfactory gum solvent.

(*b*) Bring the bomb and the gasoline to be tested to a temperature of 60 to 80 F. (15.6 to 26.7 C.). Place the glass liner containing the catalyst (Note 7) in the bomb and add 100 ± 1 ml. of sample. Close the bomb and introduce oxygen until a pressure of about 100 psi. is obtained. Allow the gas in the bomb to escape slowly in order to flush out the air originally present. Introduce oxygen again until a pressure of about 100 to 102 psi. is obtained, and then observe for leaks (Note 8). If there are no leaks, the test may be continued.

NOTE 7.—The catalyst shall be sandblasted to a uniform gray color, washed in HCl (2 per cent), rinsed with distilled water and then with acetone, and stored in kerosine.

NOTE 8.—An initial rapid drop in pressure, generally not over 6 psi. may be observed because of the solution of oxygen in the sample. If after a few minutes the rate of pressure drop does not exceed 1 psi. in 10 min., the absence of leaks may be assumed.

(*c*) Place the charged bomb in the bath of vigorously boiling water, being careful to avoid shaking, and record the time of immersion as the starting time. Maintain the temperature of the water at 212 ± 0.2 F. (100 ± 0.11 C.). Leave the bomb in the oxidation bath for 5 hr. (Note 9). If the atmospheric pressure is such that the temperature of boiling water is not within the specified range of 212 ± 0.2 F. (100 ± 0.11 C.) but is or can be made to fall within the range of 209 to 215 F. (98.3 to 101.7 C.), increase or decrease the time of heating from 5 hr. to within plus or minus 5 min. of the equivalent time at the operating temperature in accordance with the schedule given in Table I.

TABLE I.—AGING TIME AT VARIOUS TEMPERATURES EQUIVALENT TO 5 HR. AT 212 F. (100 C.).

Temperature		Aging Time	
Deg. Cent.	Deg. Fahr.	Hr.	Min.
98.4	209.1	5	48
98.6	209.5	5	41
98.8	209.8	5	35
99.0	210.2	5	29
99.2	210.6	5	23
99.4	210.9	5	17
99.6	211.3	5	11
99.8	211.6	5	6
100.0	212.0	5	0
100.2	212.4	4	54
100.4	212.7	4	49
100.6	213.1	4	44
100.8	213.4	4	39
101.0	213.8	4	34
101.2	214.2	4	29
101.4	214.5	4	24
101.6	214.9	4	20

NOTE 9.—If, as indicated by a steady drop in pressure, a leak develops after the bomb is placed in the bath, the test shall be discarded.

(*d*) At the completion of the oxidation period, remove the bomb from the bath and cool rapidly with water while the charging valve is still closed. Release the pressure slowly, take the bomb apart, and remove the glass liner.

(*e*) Filter the oxidized gasoline through a clean, dried, weighed Gooch crucible and save the filtrate. Wash the interior of the liner and the catalyst twice with fresh 10-ml. portions of solvent (Note 6) to remove any insoluble gum or visible lead precipitate. Filter the washings through the crucible and add the filtrate to the filtered oxidized sample. To insure homogeneity, mix the combined filtrates thoroughly and reserve for the gum determination (Paragraph (*g*)). Wash any visible precipitate remaining on the catalyst or in the glass liner with additional solvent. Collect the precipitate in the crucible, but discard the wash liquid. Dry the crucible at 212 F. (100 C.) in an oven for 1 hr., cool in a desiccator, and weigh. Record the increase in weight as *visible lead precipitate.*

NOTE 10.—Avoid exposing the sample to daylight prior to and during this test. At the end of the test no visible precipitate shall be present in the glass liner.

(*f*) Wash two test beakers, and a similar tare beaker, free from gum with the solvent (Note 6), immerse for at least 12 hr. in chromic-acid cleaning solution, and wash thoroughly, first with tap water and then with distilled water. Subsequent to removal from the cleaning solution, handle the beakers only by means of clean metal forceps. After washing, thoroughly dry all three beakers by heating to about 302 F. (150 C.), and allow to cool simultaneously for the same period of time, but not less than 1 hr., in a desiccator. Do not use a drying agent in either the balance case or the desiccator. Weigh each test beaker, using the tare (Note 11) on the opposite balance pan. After weighing, replace the tare beaker in the desiccator and place the test beakers in the wells of the bath as shown in Fig. 2. Adjust the air flow to a rate, at the point of measurement, of 1 liter per sec. ± 15 per per cent. Heat the bath so that the temperature of the effluent air, measured by a thermometer placed on the bottom of the beaker at the center, is from 305 to 320 F. (151.7 to 160 C.). This is governed largely by the rate of boiling of the bath liquid and the rate of air flow.

NOTE 11.—For routine tests it is permissible to use the same beaker as a tare for more than one test beaker but not for more than six test beakers, provided all are heated at the same time.

(*g*) Remove the conical adapters and pour approximately one half (plus or minus 2 ml.) of the reserved filtrate (Paragraph (*e*)) into each beaker; then replace the adapters on the air outlet so that they are vertically above the center of the surface of the liquid. After the liquid has evaporated, which should require from 5 to 10 min., leave the beakers in place for an additional 15 min. the air supply being maintained. During this 15-min. period, heat the tare beaker to about 302 F. (150 C.). Allow both test and tare beakers to cool simultaneously for the same period of time, but not less than 1 hr., in a desiccator under the same conditions as prescribed in Paragraph (*f*). Weigh the two test beakers as before the test (Note 11) and determine the combined increase in weight of the beakers.

(*h*) If the gasoline contains tetraethyl lead and the increase in weight found under Paragraph (*g*) exceeds 5.0 mg. per 100 ml., treat the residue of each beaker as follows: Add 10 ml. of H_2SO_4 (approximately 95 per cent) to the residue and evaporate the solution to strong fumes of H_2SO_4. Oxidize any organic material by cautiously adding HNO_3 drop by drop until a water-white solution is obtained. Wash the solutions from both test beakers into a single 400-ml. beaker and add 0.2 to 0.3 g. of $(NH_4)_2SO_4$ to remove completely any HNO_3 that may be present. Fume the combined solutions until only 6 to 8 ml. of solution remain. Cool, dilute to 70 ml., and boil gently for 5 min. on a hot plate. Allow the contents of the beaker to cool to room temperature, add 70 ml. of alcohol, and allow the solution to stand for at least 4 hr. Filter the solution through a clean, dried Gooch crucible, wash the precipitate, first with cold H_2SO_4 (2 per cent) and then with alcohol, dry at 110 to 120 C., and weigh as $PbSO_4$. Multiply this weight by 0.85 and subtract the result from the increase in weight of the two beakers as determined in Paragraph (*g*) to obtain the weight of residue corrected for tetraethyl lead.

Report

4. (*a*) Report the value determined in Section 3 (*g*) as the amount of potential gum per 100 ml. of sample.

(*b*) For samples tested in accordance with Section 3 (*h*), report the value determined in Section 3 (*h*) as the potential gum per 100 ml. of sample, corrected for tetraethyl lead.

APPENDIX IV

PROPOSED METHOD OF TEST FOR OXIDATION CHARACTERISTICS OF HEAVY-DUTY CRANKCASE OILS[1, 2]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia, Pa.

Scope

1. (*a*) This method of test is intended for use in determining the oxidation and bearing-corrosion characteristics (Note 1) of engine crankcase oils designed for use under heavy-duty service conditions.

(*b*) In this method of test a crankcase lubricant is evaluated only in respect to stability or resistance to oxidation, bearing corrosion, and the deposition of intrinsic contaminants resulting from decomposition, oxidation, or other changes that occur in the lubricant in service (Note 2).

(*c*) Other characteristics of a lubricant intended for heavy-duty service, such as freedom from the tendency to cause ring-sticking, valve-sticking, or combustion-chamber deposits, are not evaluated by this test method. The ability of the crankcase lubricant to hold in suspension extrinsic contaminants is not determined in this engine oxidation test. The so-called "detergent" characteristics of a lubricant, which must be considered in connection with lubricants intended for use in compression-ignition engines, are not evaluated by this method of test.[3]

NOTE 1.—In evaluating crankcase oils for heavy-duty service, several factors, in addition to the conventional inspection data, must be considered; namely, stability or resistance to oxidation, resistance to bearing corrosion, and prevention of engine deposits.

NOTE 2.—The depreciation of an oil in service results from contamination from two major sources:

(*1*) Sources external to the lubricating system. These extrinsic contaminants, including products of combustion from the combustion zone, depend upon the design, mechanical condition, and specific conditions under which the engine is operated. Extrinsic contaminants may vary in their effect upon the lubricating oil and its final condition.

(*2*) Sources inherent in the lubricating system and in the crankcase oil. These intrinsic contaminants result from changes in the oil and therefore depend upon the type and quality of the crankcase oil as well as upon the operating conditions.

APPARATUS

Apparatus

2. The engine described in Appendix I shall be used without modification for determining oxidation and bearing-corrosion characteristics of heavy-duty crankcase oils.

Reference Oils

3. Reference oils[4] shall be employed for standardizing engine operation and for bracketing the crankcase oil under test.

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

[2] Published as information, June, 1942.

[3] The Caterpillar series of tests is recommended for determining "detergent" characteristics under engine operating conditions.

[4] The use of a group of oils, on which performance in service has been established, as reference oils for bracketing unknown oils is discussed in detail in papers by H. C. Mougey and J. A. Moller, "The Testing of Heavy-Duty Motor Oils," *Proceedings*, Am. Petroleum Inst., Vol. 22, Part III, pp. 100 to 121 (1941); also, *Journal*, Soc. Automotive Engrs., vol. 50, No. 10, October, 1942.

Standard Operating Conditions

4. The engine shall be operated under the following standard conditions:

(*a*) *Engine Speed.*—3150 ± 25 rpm.

(*b*) *Load.*—30 ± 1 hp. (uncorrected).

(*c*) *Total Operating Time.*—36 hr. (Note 1) (see Section 12(*e*)).

NOTE 1.—The test may be terminated in less than 36 hr. if excessive oxidation develops.

(*d*) *Spark Advance.*—52 ± 5 deg.

(*e*) *Coolant Outlet Temperature.*—200 ± 2 F.

Coolant Inlet Temperature, min.—190 F.

(*f*) *Oil Temperature*:

S.A.E. 30 grade	280 ± 2 F.
10W or S.A.E. 10 grade	265 ± 2 F.

(*g*) *Air-Fuel Ratio.*—14.5 ± 0.5 to 1.

(*h*) *Cam Angle.*—39 deg. If cam angle cannot be measured, set breaker-point gap to 0.018 in.

(*i*) *Spark-Plug Gap.*—0.040 in.

(*j*) *Valve Clearances, Hot* (Note 2):

Inlet	0.010 in.
Exhaust	0.020 in.

NOTE 2.—The valve clearance shall be checked at the start and again at 16 hr. and at the end of the 36-hr. test period.

(*k*) *Exhaust Back Pressure.* — 1.0 ± 0.5 in. of mercury.

PROCEDURE

Run-In of New Engine

5. A new engine should be run-in in accordance with the run-in schedule described in Appendix II.

Cleaning

6. Engine parts should be thoroughly cleaned prior to each test in accordance with the cleaning schedule described in Appendix III.

Reconditioning

7. (*a*) The engine shall be thoroughly reconditioned after each run. All parts shall be inspected, and defective or worn parts shall be replaced.

(*b*) The engine shall be maintained so that the oil consumption shall be within the following limits:

S.A.E. 30 grade	0.07 to 0.12 qt. per hr.
10W or S.A.E. 10 grade	0.08 to 0.15 qt. per hr.

Piston Rings

8. New piston rings shall be installed prior to each test run.

Bearings

9. (*a*) Main bearings and babbitt connecting-rod bearings shall be adjusted to the clearance prescribed in Appendix I.

(*b*) Copper-lead test bearings should be installed initially in connecting rods Nos. 3 and 4 and shall be fitted within the clearance prescribed in Appendix I. After each test run the copper-lead bearings should be shifted to connecting rods Nos. 1 and 6, or Nos. 2 and 5 and the cycle continued, to minimize wear and early replacement of the crankshaft.

(*c*) The copper-lead bearing halves shall be weighed individually on an analytical balance to the nearest 0.01 g. before being installed in the engine.

Fuel

10. (*a*) The fuel used in the engine shall conform to the following requirements:

Test	Requirement	A.S.T.M. Designation[a]
Octane number, min.	72	D 357
Sulfur, max., per cent	0.04	D 90
Lead, ml. of tetraethyl lead per gal.	2.5 to 3.0	D 526

[a] These designations refer to the following methods of the American Society for Testing Materials:
Tentative Method of Test for Knock Characteristics of Motor Fuels (A.S.T.M. Designation: D 357),[5]
Tentative Method of Test for Sulfur in Petroleum Oils by Lamp Method (A.S.T.M. Designation: D 90),[6] and
Standard Method of Test for Tetraethyl Lead in Gasoline (A.S.T.M. Designation: D 526).[7]

(*b*) Fuel from the same source of supply shall be used in any series of tests in which a crankcase oil is being compared or bracketed with a group of reference oils.

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[5] 1942 Book of A.S.T.M. Standards, Part III, p. 935.

[6] *Ibid.*, p. 977.

[7] *Ibid.*, p. 219.

Run-In Schedule for Reconditioned Engine

11. (*a*) After reassembling, the engine shall be run-in, prior to each test run, in accordance with the following schedule:

(*1*) Fill crankcase with a highly refined, uninhibited and uncompounded, mineral oil having a high viscosity index (S.A.E. 20 grade).
(*2*) Run 1 hr. at approximately 1000 rpm. with approximately 5 hp. output.
(*3*) Run 1 hr. at approximately 1500 rpm. with approximately 10 hp. output.
(*4*) Run 2 hr., gradually increasing speed and load to approximately 2200 rpm. and 25 hp. output.
(*5*) Drain crankcase and refill with the crankcase lubricant under test.
(*6*) Run 20 min. at 2000 rpm. with 15 to 20 hp. output.
(*7*) Run 40 min. at 2000 rpm. with 25 hp. output.
(*8*) Run 3 hr. at 3000 rpm. with 30 hp. output.
(*9*) Drain crankcase.

(*b*) During the run-in period the coolant and crankcase-oil sump temperatures shall be maintained within the following limits:

Coolant outlet temperature	170 ± 5 F.
Oil temperature, max.	225 F.

Operation of Engine during Test

12. (*a*) The crankcase shall be filled with 4 qt. of the crankcase lubricant to be tested (Note).

NOTE—The original 4-qt. of oil used and the 1-pt. additions specified during the test period may be made on a weight basis.

(*b*) The standard operating conditions prescribed in Section 4 shall be observed.

(*c*) The engine shall be operated continuously for not less than 8 nor more than 16 hr. in any one test period. The engine shall be stopped and remain idle for not less than 8 hr. between test periods. A test period of less than 8 hr. may be employed at the start and at the end of the 36-hr. test period.

(*d*) At the start of each test period the engine shall be run for 10 min. at 2000 rpm. with 15 to 25 hp. output, or until the crankcase-oil sump temperature reaches 150 F. The speed shall then be increased to 3150 rpm.

(*e*) The test period shall be determined from the time the crankcase-oil sump temperature reaches 280 F. for the S.A.E. 30 grade and 265 F. for the 10W or S.A.E. 10 grade, and shall be timed only for the periods during which the engine is operated at the prescribed crankcase-oil sump temperatures.

(*f*) The crankcase oil shall not be drained during the 36-hr. test period.

(*g*) One pint of fresh oil shall be added to the crankcase every 4 hr. during the 36-hr. test period. The addition of 1 pt. of oil at the 8-hr., 16-hr., and 24-hr. intervals shall be made as required in Paragraph (*h*).

(*h*) At the 8-hr., 16-hr., and 24-hr. intervals, two ½-pt. samples shall be withdrawn from the oil-sampling cock. The first ½-pt. sample withdrawn shall be returned to the crankcase. The second ½-pt. sample shall be retained as the sample for analysis. After withdrawing these samples, the engine shall be shut down and allowed to stand 1 min., at which time the level of oil in the crankcase shall be measured by the dipstick. If the oil level is less than 3½ qt., fresh oil shall be added to bring the level to 3½ qt. If the oil level is more than 3½ qt., the engine shall be started and the excess amount of oil above 3½ qt.

withdrawn through the sampling cock and the engine then stopped. After adjusting the oil level to 3½ qt., 1 pt. of fresh oil shall be added as required in Paragraph (*g*) and the test continued.

(*i*) The 36-hr. sample shall be withdrawn, at the conclusion of the test, in the same manner as the 8-hr., 16-hr., and 24-hr. samples.

(*j*) The following data shall be recorded at hourly intervals during the 36-hr. test period:

(*1*) Engine speed in revolutions per minute,
(*2*) Beam load in pounds (report as horsepower),
(*3*) Crankcase-oil sump temperature in degrees Fahrenheit,
(*4*) Oil pressure in pounds per square inch,
(*5*) Coolant temperatures, at outlet and at inlet, in degrees Fahrenheit,
(*6*) Exhaust back pressure in inches of mercury, and
(*7*) Intake manifold depression in inches of mercury.

Air-Fuel Ratio

13. (*a*) The air-fuel ratio shall be determined at the start and again at the end of the 36-hr. test period in accordance with the procedure described in Appendix IV.

(*b*) The specific fuel consumption or the time required for the consumption of 300 ml. of fuel shall be recorded at the time samples of exhaust gas are withdrawn for the determination of the air-fuel ratio.

Inspection of Engine Parts

14. (*a*) At the conclusion of the test, the engine shall be torn down and inspected. The following parts shall be rated with respect to varnish and sludge deposits:

Varnish deposits:
  Piston skirts,
  Cylinder walls,
  Rocker-arm cover,
  Push-rod cover plate, and
  Crankcase oil pan.
Sludge deposits:
  Rocker-arm assembly,
  Rocker-arm cover,
  Push-rod cover plate,
  Oil screen, and
  Crankcase oil pan.

(*b*) The engine parts enumerated in Paragraph (*a*) shall be rated on a scale from 10 to 0. A rating of 10 shall be assigned to parts free of deposits. A rating of 0 shall be assigned to parts on which the deposits have reached a maximum or have actually caused engine failure.

(*c*) Photographs (Note 1) shall be made of the following engine parts:

Two pistons (Note 2),
Rocker-arm assembly,
Rocker-arm cover,
Push-rod cover plate, and
Oil screen.

NOTE 1.—Kodachrome positives of typical pistons may be employed for recording character and extent of piston deposits. Photomicrographs may be employed for recording extent of copper-lead bearing corrosion.[8]

NOTE 2.—If it is necessary to use phosphate-coated pistons, photographs will not show the extent of varnish formation on the piston skirts. In this case it will be necessary to wash the piston thoroughly with gasoline in order to remove excess oil, and to remove varnish from piston skirts by dissolving it in acetone. The varnish deposit should be reported in milligrams of the acetone-soluble material per piston.

(*d*) The copper-lead bearing halves shall be weighed to the nearest 0.01 g. to determine loss due to action of corrosive oxidation products. If visual inspection of the bearing halves shows evidence of mechanical failure, the loss in weight shall not be included in the report of bearing loss.

Inspection of Oil Samples

15. (*a*) The following samples shall

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[8] H. L. Grange, "Metallographic Preparation of Copper-Lead Bearings," *Metal Progress*, Vol. 30, No. 5, November, 1940, p. 674.

be examined to determine the rate of oxidation during the 36-hr. test period:

New oil,
16-hr. sample (Section 12 (*h*)), and
36-hr. sample (Section 12 (*i*)).

(*b*) If the test is stopped prior to the completion of the 36-hr. test period, the 8-hr. sample (Section 12 (*h*)) shall be examined.

(*c*) The samples prescribed in Paragraphs (*a*) and (*b*) shall be examined by the following methods of test:

Test	A.S.T.M. Designation[a]
Neutralization number	D 663
Naphtha-insoluble matter (method A or method B)	[b]
Chloroform-soluble matter (method A) or asphaltenes (method B)	[b]
Conradson carbon residue	D 189
Ramsbottom carbon residue (additional optional test)	D 524
Ash	D 482
Viscosity, Saybolt Universal, at 100 F.	D 88

[a] These designations refer to the following methods of the American Society for Testing Materials:

Tentative Method of Test for Neutralization Number of Petroleum Products by Color-Indicator Titration (A.S.T.M. Designation: D 663),[9]

Standard Method of Test for Carbon Residue of Petroleum Products (Conradson Carbon Residue) (A.S.T.M. Designation: D 189),[10]

Standard Method of Test for Carbon Residue of Petroleum Products (Ramsbottom Carbon Residue) (A.S.T.M. Designation: D 524),[10]

Tentative Method of Test for Ash Content of Petroleum Oils (A.S.T.M. Designation: D 482),[11] and

Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (A.S.T.M. Designation: D 88).[12]

[b] See Appendix V.

Report

16. (*a*) The report shall include the following:

(*1*) Brand name or experimental designation of crankcase lubricant tested,
(*2*) Log sheet incorporating data required in Section 12 (*j*)),
(*3*) Oil consumption in quarts per hour,
(*4*) Air-fuel ratio, including determinations of carbon dioxide, oxygen, and carbon monoxide,
(*5*) Specific fuel consumption or time required for consumption of 300 ml. of fuel,
(*6*) Evaluation of engine parts enumerated in Section 14 (*a*),
(*7*) Photographs of engine parts enumerated in Section 14 (*c*),
(*8*) Copper-lead bearing weight loss reported as loss for each individual bearing half,
(*9*) Results of examination of the oil samples designated in Section 15 (*a*) and (*b*) by the methods of test specified in Section 15 (*c*).

(*b*) A suggested log sheet and report form are shown in Appendices VI and VII.

APPENDIX I

APPARATUS

Apparatus:

A1. The apparatus described in this appendix shall be used without modification for determining the oxidation and bearing-corrosion characteristics of crankcase oils when operating under heavy-duty service conditions. The apparatus shall consist of a spark-ignition internal-combustion engine[13] together with suitable loading and accessory equipment.

Dimensional Specifications:

A2. *Engine.*—A spark-ignition internal-combustion engine, with dimensions as follows:

Number of cylinders	6 in line
Compression ratio	6.5 to 1
Bore, in.	3½ (nominal)
Stroke, in.	3¾ (nominal)
Displacement, cu. in.	216.5

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[9] 1942 Book of A.S.T.M. Standards, Part III, p. 954.

[10] *Ibid.*, pp. 102, 106.

[11] *Ibid.*, p. 898.

[12] *Ibid.*, p. 248.

[13] At present (June, 1942), the sole authorized manufacturer of the oxidation test engine is the Chevrolet Motor Division, General Motors Corporation. Engines, complete with fuel and electrical systems, but without clutch, transmission and battery, may be secured by laboratories equipped for research on lubricants from the Engineering Department, Chevrolet Motor Division, on approval of order by H. C. Mougey, chairman of Technical Committee B, of Committee D-2, R 10-112, General Motors Research Bldg., Detroit, Mich.

Valve-port diameter, throat, in.:	
inlet	$1\frac{1}{4}$
exhaust	$1\frac{13}{16}$
Connecting-rod bearings:	
material	babbitt (centrifugally cast)
diameter, in.	2.3135 to 2.3140
length, in.	$1\frac{1}{2}$
clearance	selective fit
Piston-pin bushings:	
material	cast bronze
diameter, inside	slip fit on pin
length, in.	$\frac{15}{16}$
Front main bearing:	
material	removable steel-backed babbitt
diameter, in.	2.6865 to 2.6875
length, in.	$1\frac{3}{16}$
No. 2 main bearing:	
material	removable steel-backed babbitt
diameter, in.	2.7175 to 2.7185
length, in.	$1\frac{3}{16}$
No. 3 main bearing:	
material	removable steel-backed babbitt
diameter, in.	2.7485 to 2.7495
length, in.	$1\frac{7}{16}$
Rear main bearing:	
material	removable steel-backed babbitt
diameter, in.	2.7795 to 2.7805
length, in.	$1\frac{5}{8}$
Connecting rod, center to center, in.	$6\frac{13}{16}$
Pistons:	
material	cast alloy iron
surface treatment	tin plating or phosphate coating
skirt clearance	selective fit
Piston rings:	
Compression:	
material	cast alloy iron
type	plain
number per piston	2
width, in.	0.1235 to 0.1240
wall thickness, max., in.	0.155
gap clearance, in.	0.005 to 0.015
groove clearance, in.	0.0015 to 0.003
Oil control:	
material	cast alloy iron
type	drilled
number per piston	1
width, in.	0.1860 to 0.1865
wall thickness, max., in.	0.155
gap clearance, in.	0.005 to 0.015
groove clearance, in.	0.002 to 0.0035
Spark plugs:	
type	AC 104 or Champion Y-5
size, mm.	10

Fan Blades and Ventilating System:

A3. (*a*) The fan blades, bolted directly to the water pump, shall be removed.

(*b*) The crankcase shall be permitted to breathe "normally". No connection shall be made to the engine-crankcase ventilating system that will decrease or increase normal crankcase breathing.

Cooling System:

A4. The cooling system may be equipped with automatic temperature-control apparatus and mixing valves for holding the coolant temperature within the specified temperature range. In localities where the water supply contains solids, in suspension or solution, that have a tendency to precipitate in the engine water passages and change the heat flow to the coolant, a closed system employing a submerged car or truck radiator may be used as a heat exchanger. Care must be exercised, when hard or aerated water is used for cooling, to check pump impellers for erosion.

Exhaust System:

A5. An exhaust pipe shall be attached to the engine exhaust manifold. The use of a short straight-through muffler with passage equal in diameter to that of the exhaust pipe is permissible. The use of a multiple exhaust system is permissible, provided a back pressure of 1.0 ± 0.5 in. of mercury is maintained on each individual exhaust manifold.

Power-Absorbing Unit:

A6. The engine shall be connected to an electric generator or a power-absorbing unit capable of maintaining the prescribed engine speed (Section 4 (*a*)). If a calibrated propeller is used for absorbing the power output, it shall be mounted on a separate shaft and in such a manner that it will not interfere with the normal cooling of the engine nor with the normal functioning of the crankcase ventilating system.

Instruments and Accessories:

A7. (*a*) In addition to the conventional instrumentation required for dynamometer test work, a pipette for determining fuel consumption and suitable manometers for measuring intake-manifold vacuum and exhaust-manifold pressure shall be provided.

(*b*) The connection for the manometer for determining exhaust back pressure shall be a standard $\frac{1}{8}$-in. pipe inserted in the exhaust manifold. The pipe shall be flush with the inner wall of the manifold and shall be so located that the center line of the $\frac{1}{8}$-in. pipe is $\frac{3}{4}$-in. from the gasket face of the manifold flange connecting to the exhaust pipe.

(*c*) The manometer for determining intake manifold vacuum shall be connected to the windshield-wiper fitting provided on the intake manifold.

(*d*) An oil-sampling valve shall be attached to the oil gallery.

(*e*) Thermocouples shall be provided for determining coolant temperature "in" and "out" of the engine cooling system.

(*f*) A thermocouple for determining crankcase-oil sump temperature shall be located in the crankcase at the exact depth and position of the lower end of the dip-stick.

(*g*) In order to maintain the crankcase-oil sump temperature within the specified limits, it is necessary either to heat or to cool the crankcase oil during the major part of the run-in and 36-hr. test periods. Strip heaters attached to the crankcase-oil pan, and immersion heaters and water-cooling coils installed in the oil pan, shall not be employed for this purpose. Radiant or glow heaters enclosed in a metal jacket mounted below the oil pan may be used for heating the crankcase oil. A blower discharging through this same metal jacket may be used for cooling the crankcase oil. The metal jacket, radiant heaters, and blower discharge should be arranged so that the heat transfer shall be through the lower part of the oil pan rather than through the side walls of the oil pan or through the walls of the crankcase. A metal pan may be welded to the lower part of the oil pan to provide a jacket for steam-heating or water-cooling the crankcase oil.

Inspection and Maintenance of Apparatus:

A8. It is recommended that a systematic inspection of the testing apparatus be carried out with the utmost care after each 36-hr. test period. This inspection shall include the following:

(*a*) *Fuel System.*—Any foreign matter in the fuel containers, float bowls, lines, and carburetor should be removed. Blowing with air and then flushing the fuel system with gasoline and benzol is a convenient method.

(*b*) *Breaker Points.*—If necessary, pits should be removed and breaker-point clearance adjusted, or new points installed.

(*c*) *Spark Advance.*—The spark advance setting should be checked and reset if necessary. If the automatic (centrifugal) and vacuum advance will not maintain the spark advance within the prescribed limits, a manual spark advance may be installed.

(*d*) *Spark Plugs.*—The porcelain should be carefully inspected for cracks, and defective plugs replaced. The spark gap should be carefully checked, and adjusted if necessary. When plugs are re-used, any carbon deposit on the metal body of the plug and on the electrodes should be removed mechanically before making gap adjustments.

(*e*) *Combustion Chamber.*—Carbon deposits should be removed by scraping.

(*f*) *Pistons and Rings.*—Carbon should be removed from the ring grooves and from both sides of the crown.

New pistons should be fitted in accordance with the procedure described on pages 6-3 and 6-4 of the 1942 Chevrolet Shop Manual.[14]

New piston rings should be fitted in accordance with the procedure described on pages 6-5 and 6-6 of the 1942 Chevrolet Shop Manual.[14]

In inserting the piston into the cylinder the rings should be compressed with a "piston inserter" until they enter the cylinder freely. The chamfer on the bottom of the inserter should fit the chamfer at the top of the cylinder. Extreme care should be exercised to prevent forcing the rings into the cylinder bore.

(*g*) *Cylinder Wear.*—The cylinder block should be replaced or rebored when it shows wear or out-of-roundness in excess of 0.003 in., becomes scored or scuffed, or loses compression.

(*h*) *Compression Pressure.*—A change of 15 per cent in compression pressure shall be considered abnormal and should be corrected.

(*i*) *Valves.*—Valves shall be removed and lapped or reground after each test period.

(*j*) *Cooling System.*—The cooling system should be inspected frequently and kept clean and free of leaks.

(*k*) *Torque Wrench Settings.*—Uniform torque wrench settings should be employed as follows:

	Torque, ft-lb.
Spark plugs	15
Main bearings	100
Connecting-rod bearings	45 to 50
Cylinder head	75
Rocker-arm shaft support	25 to 30

Bearings:

A9. (*a*) The main bearings shall be adjusted in accordance with the procedure described on pages 6–9 and 6–10 of the 1942 Chevrolet Shop Manual.[14]

(*b*) The babbitt connecting-rod bearings shall be adjusted in accordance with the procedure described on page 6–18 of the 1942 Chevrolet Shop Manual.[14]

(*c*) The special copper-lead connecting-rod bearings[15] shall be installed in rods Nos. 3 and 4, as follows:

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[14] Copies of the 1942 Chevrolet Shop Manual may be obtained from the Chevrolet Motor Division, General Motors Corp., Detroit, Mich., at 25 cents each.

(*1*) The special connecting rods[16] and copper-lead test bearings are machined to give a clearance of from 0.0025 to 0.0035 in. No means for adjustment is provided. When shifting connecting rods as outlined in Section 9(*b*), the connecting rods only shall be moved to the new positions; the pistons shall remain in their original positions.

(*2*) The following method is suggested for determining the clearance of the copper-lead bearings:

Install the piston in the cylinder and properly align the rod and rod bearing in contact with the journal. Place a strip of pure lead approximately $\frac{1}{16}$ in. in width by 0.005 to 0.006 in. in thickness, previously coated with a thin film of light oil, on the cap bearing in contact with and parallel to the axis of the journal. Install cap and connecting rod dipper and tighten nuts with a torque wrench (45 to 50 ft-lb.).

Oscillate crankshaft through approximately 30 deg. to flow the lead. Remove cap and lead strip. If the clearance, as indicated by the thickness of the lead strip, is within the limits specified in Item 1 above, reassemble and tighten nuts, using the same torque as before.

With less than the minimum clearance specified, the lead strip may leave an impression on the bearing surface. If this occurs, the thickness of the lead strip is not an accurate indication of bearing clearance

The out-of-round condition of the journal may be determined by inserting lead strips at the points of minimum and maximum clearance. The crankshaft should be replaced when the connecting-rod journals develop an out-of-round condition of more than 0.002 in.

APPENDIX II

RUN-IN SCHEDULE

Run-in Schedule:

A10. (*a*) The run-in schedule shown in Table I is recommended for breaking-in a new engine.

NOTE.—This recommended schedule is required for a new engine only. The run-in schedule for breaking-in new rings is described in Section 11.

(*b*) The coolant temperature shall be held at 170 ± 5 F. The crankcase-oil sump temperature should not exceed 225 F.

(*c*) The recommended speeds and loads are shown in Table I.

TABLE I.—RUN-IN SCHEDULE FOR NEW ENGINE.
Total Run-In Time: 38 hr., 30 min.

Time	Engine Speed, rpm.	Beam Load, lb.			Brake Horsepower
		K-3000 (1.75 ft. arm)	K-4000 (1.31 ft. arm)	K-5000 (1.05 ft. arm)	
1 hr.	900	15.8	21.0	26.3	4.7
2 hr.	1350	21.0	28.0	35.0	9.5
4 hr.	1800	28.5	38.0	47.5	17.1
5 hr.	2250	38.3	51.0	63.8	25.7
7 hr.	2465	43.5	58.0	72.5	35.8
4 hr.	2600	full	full	full	...
4 hr.	3000	full	full	full	...
4 hr.	3200	full	full	full	...
Set controls at 3200 rpm. to give specific beam load and do not change during remainder of schedule..................		45	60	75	...
4 hr.	3400	Accelerate 10 times from 3400 to 3800 rpm. at end of each hour.			
3½ hr.	3600	Accelerate 10 times from 3400 to 3900 rpm. at end of each hour.			

APPENDIX III

CLEANING ENGINE PARTS

Cleaning Materials:

A11. The following cleaning materials will be required:

(*a*) *Electro-Cleaning Compound.*—The electro-cleaning compound shall have the following composition:

Sodium carbonate, per cent................	35
Trisodium phosphate, per cent.............	55
Sodium hydroxide, per cent...............	10

(*b*) *Cleaning Solution.*—The cleaning solution shall have the following composition:

Ethyl acetate, ml........................	1600
Denatured alcohol, ml..................	1200
Butyl alcohol, ml.......................	200
Cresol compound (U.S.P.), ml...........	1000

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[15] At present (June, 1942), the sole authorized manufacturer of the special copper-lead connecting rod bearing shells is The Cleveland Graphite Bronze Co., Cleveland, Ohio. The thin back bearing shells are identified as B/P 21500 and B/P 21501.

[16] At present (June, 1942), the sole authorized manufacturer of the special connecting rods machined to accommodate the copper-lead test bearings is the Research Laboratories Division, General Motors Corp., Detroit, Mich.

The cresol compound should be added to the mixture of the other solvents.

General Cleaning Procedure:

A12. (*a*) At the conclusion of the new-engine run-in schedule described in Appendix II, and at the conclusion of each test, after the parts specified in Section 14 have been examined, rated, and photographed, all engine parts shall be thoroughly cleaned.

(*b*) The rocker-arm assembly, rocker-arm cover, push rods, push-rod cover plate, complete piston assembly, cam followers, oil-pump strainer, and oil pan may be cleaned in a trichlorethylene degreaser or in a suitable metal cleaning bath.

(*c*) Following the cleaning operation described in Paragraph (*b*), the complete piston assembly and cam followers shall be given a further cleaning treatment in an electro-cleaning bath to completely remove varnish deposits. The electro-cleaning bath shall contain 0.5 oz. per gallon of water of the cleaning compound described in Section A11 (*a*). The bath shall be operated at 200 to 215 F. The parts to be cleaned shall be suspended in the bath as the cathode for 15 to 30 min. at a potential of approximately 6 v. On removal from the bath the parts should be rinsed in cold clean water.

(*d*) Following the cleaning operation described in Paragraph (*c*), the piston rings shall be removed and the piston-ring grooves shall be scraped free from deposits. The piston pin shall be removed from the piston and connecting rod and further cleaned with the cleaning solution described in Section A11 (*b*). Crocus cloth may be used, if necessary, to remove varnish deposits.

(*e*) The cylinder walls, crankshaft, interior of crankcase, and the timing-gear housing shall be washed with Stoddard solvent. The cylinder walls and crankshaft connecting-rod journals shall be further cleaned by applying the cleaning solution described in Section A11 (*b*) with a stiff bristle brush. The crankshaft connecting-rod journals may be polished with crocus cloth, if necessary, to remove varnish deposits.

(*f*) Immediately following the cleaning operations, all parts shall be sprayed with or dipped in the oil described in Section 11 (*a*), Item (*1*).

(*g*) The crankshaft and camshaft can be cleaned satisfactorily without removal from the engine unless the engine is in an unusually dirty condition.

Procedure for Cleaning Copper-Lead Bearings:

A13. (*a*) New copper-lead bearings should be inspected before weighing as specified in Section 9 (*c*). New bearings are free from oil and generally do not require extensive cleaning. If cleaning is required, bearings may be washed with acetone or chloroform and dried before weighing.

(*b*) At the completion of the test, prior to the final weighing specified in Section 14 (*d*), bearings shall be thoroughly cleaned with acetone or chloroform. Varnish on the bearing surfaces, and oil absorbed by the bearings, shall be thoroughly removed and the bearings dried before weighing.

APPENDIX IV

AIR-FUEL RATIO

Air-Fuel Ratio:

A14. The air-fuel ratio shall be calculated from the exhaust-gas analysis.

Sampling and Apparatus:

A15. (*a*) When a number of engines are connected to a common exhaust system, the sample for analysis shall be withdrawn from the exhaust manifold through the fitting provided for attachment to the manometer for determining exhaust-manifold pressure. When the engine is provided with an individual exhaust system, the sample for analysis may be withdrawn through a $\frac{1}{4}$-in. copper tube extending 3 or 4 ft. into the tail pipe.

(*b*) The exhaust gas shall be analyzed by means of an Orsat-type gas-analysis apparatus.

(*c*) A lead of 3 or 4 ft. of $\frac{1}{4}$-in. copper tubing shall be provided, between the exhaust manifold fitting or sampling tube and the gas-analysis apparatus, for cooling the exhaust-gas sample. Before collecting the sample for analysis, the connecting tube and burette shall be flushed free from air or other gas.

(*d*) The burette shall be water-jacketed and shall be filled with a 20 per cent solution of sodium sulfate in water containing 5 per cent by volume of sulfuric acid. (A small amount, 0.1 per cent, of a detergent (Dreft) may be added to the salt solution to assist in keeping the burette free of grease.)

Procedure:

A16. (*a*) Carbon dioxide, oxygen, and carbon monoxide shall be determined in the order named. The following absorbents are recommended:

Carbon Dioxide Absorbent.—Sodium hydroxide solution (25 per cent).

Oxygen Absorbent.[17]—A slightly acid solution of chromous chloride.

Carbon Monoxide Absorbent.[17]—A solution of cuprous sulfate and beta-naphthol.

(*b*) The air-fuel ratio, within the allowable range of 14.5 ± 0.5 to 1, calculated from the carbon dioxide and carbon monoxide content of the exhaust gas for a hydrocarbon fuel of the formula C_nH_{2n} is given in Table II.

(*c*) If the exhaust gas contains carbon monoxide and more than approximately 0.5 per cent oxygen, the sample may have been diluted with air, or the engine may be missing or otherwise operating in an irregular manner.

TABLE II.—VALUES OF AIR-FUEL RATIO BASED ON CARBON DIOXIDE AND CARBON MONOXIDE CONTENT OF EXHAUST GAS.

CarbonMonoxide, per cent	Carbon Dioxide, per cent														
	13.5	13.6	13.7	13.8	13.9	14.0	14.1	14.2	14.3	14.4	14.5	14.6	14.7	14.8	14.9
0.0	...	...	...	...	...	...	...	...	...	...	...	...	15.05	14.95	14.85
0.1	...	...	...	...	...	...	...	...	...	...	...	15.05	14.95	14.85	14.75
0.2	...	...	...	...	...	...	...	...	...	...	15.05	14.95	14.85	14.75	14.65
0.3	...	...	...	...	...	...	...	...	...	15.0	14.95	14.85	14.75	14.65	14.55
0.4	...	...	...	...	...	...	...	...	15.0	14.9	14.85	14.75	14.65	14.55	...
0.5	...	...	...	...	...	...	...	15.0	14.9	14.8	14.75	14.65	14.55	14.45	...
0.6	...	...	...	...	...	...	14.95	14.9	14.8	14.7	14.65	14.55	14.45	14.35	...
0.7	...	...	...	...	...	...	14.85	14.8	14.7	14.6	14.55	14.45	14.35	...	...
0.8	...	...	...	...	...	14.85	14.75	14.7	14.6	14.5	14.45	14.35	14.25	...	...
0.9	...	...	...	...	...	14.75	14.65	14.6	14.5	14.4	14.35	14.25	...	...	...
1.0	...	...	...	...	14.75	14.65	14.55	14.5	14.4	14.3	14.25	14.15	...	...	...
1.1	...	...	...	14.75	14.65	14.55	14.45	14.4	14.3	14.2	14.15	...	...	...	...
1.2	...	...	...	14.65	14.55	14.45	14.35	14.3	14.2	14.1	...	...	...	...	...
1.3	...	...	14.6	14.55	14.45	14.35	14.25	14.2	14.1	14.0	...	...	...	...	...
1.4	...	...	14.5	14.45	14.35	14.25	14.15	14.1	14.0	...	...	...	...	...	...
1.5	...	...	14.4	14.35	14.25	14.15	14.05	14.0	...	...	...	...	...	...	...
1.6	...	14.4	14.3	14.25	14.15	14.05	...	...	...	...	...	...	...	...	...
1.7	...	14.3	14.2	14.15	14.05	...	...	...	...	...	...	...	...	...	...
1.8	...	14.2	14.1	14.05	...	...	...	...	...	...	...	...	...	...	...
1.9	14.2	14.1	14.0	...	...	...	...	...	...	...	...	...	...	...	...
2.0	14.1	14.0	...	...	...	...	...	...	...	...	...	...	...	...	...
2.1	14.0	...	...	...	...	...	...	...	...	...	...	...	...	...	...

APPENDIX V[18]

INSOLUBLE MATTER IN USED CRANKCASE OILS

Method A

Scope:

A17. This method is intended for the gravimetric determination of the naphtha-insoluble matter and the naphtha-insoluble matter soluble in chloroform, in used crankcase oils.

Apparatus:

A18. The apparatus shall consist of the following:

(*a*) *Flask.*—A 300-ml. Erlenmeyer flask.

(*b*) *Gooch Crucible.*—A Gooch crucible having a capacity of 25 ml.

(*c*) *Asbestos Fiber.*—Washed and ignited asbestos fiber suitable for use in the Gooch crucible.

(*d*) *Suction Apparatus.*—A suction flask or jar with suitable means for maintaining a vacuum not to exceed 10 cm. of mercury.

(*e*) *Oven.*—An electric oven capable of maintaining a temperature of 149 ± 2.8 C. (300 ± 5 F.).

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[17] Methods of the chemists of the U. S. Steel Corp., J. M. Camp, Third Edition, pp. 38 to 40 (1927).

[18] Methods for determining insoluble matter in used crankcase oils have not yet been standardized by Committee D-2. Subcommittee X on Insoluble Content of Used Oils, of Committee D-2, is conducting cooperative work on this subject at the present time. The methods in Appendix V are suggested for use in connection with the engine oxidation test until tentative methods are issued by the Society.

Methods A and B should yield substantially the same results on naphtha-insoluble matter. Method A covers the determination of the naphtha-insoluble matter soluble in chloroform. Method B covers the determination of the chloroform-insoluble matter in the original sample. The naphtha-insoluble matter soluble in chloroform obtained in method B by subtracting the chloroform-insoluble matter from the naphtha-insoluble matter is a measure of the total asphaltenes present in the used oil. The naphtha-insoluble matter soluble in chloroform determined in method A is a measure of the asphaltenes that remain soluble in chloroform on heating at 149 C. (300 F.) for 30 min.

(*f*) *Balances.*—An analytical balance sensitive to 0.1 mg. for weighing the Gooch crucible; a laboratory balance sensitive to 0.1 g. for weighing the oil sample.

(*g*) *Desiccator.*

Reagents:

A19. (*a*) *A.S.T.M. Precipitation Naphtha.*—The A.S.T.M. precipitation naphtha[19] shall conform to the following requirements:

Test	Requirement	A.S.T.M. Designation[a]
Gravity, deg. A.P.I. at 60 F	70 to 73	D 287
Aniline point	58 to 60 C.	D 611
Initial boiling point	not less than 122 F. (50 C.)	D 86
50 per cent point	158 to 176 F.. (70 to 80 C.)	D 86
End point	not more than 266 F. (130 C.)	D 86

[a] These designations refer to the following methods of the American Society for Testing Materials:

Standard Method of Test for Gravity of Petroleum and Petroleum Products by Means of the Hydrometer (A.S.T.M. Designation: D 287),[20]

Tentative Method of Test for Aniline Point of Petroleum Products (A.S.T.M. Designation: D 611),[21] and

Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (A.S.T.M. Designation: D 86).[22]

(*b*) *Chloroform, U. S. P.*

Procedure:

A20. (*a*) Thoroughly agitate the sample until homogeneous and until all sediment is in suspension.

(*b*) Weigh 10 ± 0.1 g. of the prepared sample into a 300-ml. Erlenmeyer flask. Add 100 ml. of petroleum naphtha. Thoroughly agitate the mixture until homogeneous and allow to stand for 3 hr.

(*c*) Suspend the asbestos fiber in distilled water. Add the asbestos suspension to the Gooch crucible, apply suction, and press the asbestos into a compact mat. Build the mat to approximately 1 in. in thickness. Dry and ignite. Cool in a desiccator and weigh to 0.1 mg.

(*d*) Filter the contents of the Erlenmeyer flask through the weighed Gooch crucible. Transfer all insoluble matter from the flask to the crucible by washing with A.S.T.M. precipitation naphtha. Wash the insoluble matter in the crucible with 100 ml. of precipitation naphtha. Dry in the oven at 149 ± 2.8 C. (300 ± 5.0 F.) for 30 min. (Note). Cool in a desiccator and weigh to 0.1 mg.

NOTE.—The solubility in chloroform of the residue from the determination of naphtha-insoluble matter is affected by the time and temperature of drying. For this reason, in order to secure check results in the determination of naphtha-insoluble matter soluble in chloroform, the drying time and temperature, especially in the determination, of naphtha-insoluble matter, should be carefully controlled.

(*e*) Wash the residue from the determination of naphtha-insoluble matter (Paragraph (*d*)) with chloroform until the filtrate is colorless (100 ml. of chloroform is generally sufficient). Allow each addition of chloroform to remain on the filter pad for a few minutes before applying suction. Dry in the oven at 149 ± 2.8 C. (300 ± 5.0 F.) for 30 min. Cool in a desiccator and weigh to 0.1 mg.

Calculations:

A21. Calculate the percentages of naphtha-insoluble matter and of naphtha-insoluble matter soluble in chloroform, as follows:

$$\text{Naphtha-insoluble matter, per cent} = 10(B - A)$$

$$\text{Naphtha-insoluble matter soluble in chloroform, per cent} = 10(B - C)$$

where:

A = weight in grams of prepared, dried Gooch crucible,

B = weight in grams of Gooch crucible and contents after extraction with petroleum naphtha, and

C = weight in grams of Gooch crucible and contents after extraction with chloroform.

Method B

Scope:

A22. This procedure is intended for the gravimetric determination of the naphtha-insoluble matter and the chloroform-insoluble matter in used lubricating oils, but it may also be used for the determination of insoluble matter in sludges, engine deposits, filter cakes, etc. This same procedure may be used for the determination of matter insoluble in other solvents such as benzol, pentane, and heptane.

Apparatus:

A23. The apparatus shall consist of the following:

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[19] These requirements for precipitation naphtha are identical with those prescribed in Section 4 of the Standard Method of Test for Precipitation Number of Lubricating Oils (A.S.T.M. Designation: D 91) of the American Society for Testing Materials, 1942 Book of A.S.T.M. Standards, Part III, p. 196.

[20] 1942 Book of A.S.T.M. Standards, Part III, p. 167.

[21] *Ibid.*, p. 896.

[22] *Ibid.*, p. 131.

(*a*) *Filtration Apparatus.*—A Gooch filter and accessory equipment as shown in Fig. 1. The Gooch crucible shall have the approximate dimensions shown and shall have a porous glass filter plate having pore diameters of 10 to 35 microns. The adapter, 250-ml. Erlenmeyer flask, and vacuum jar may be of any suitable design, but the style shown in Fig. 1 is preferable if the filtrate is to be retained for additional examination. A jet aspirator or any suitable source of reduced pressure that is capable of holding an absolute pressure of 10 cm. of mercury is required.

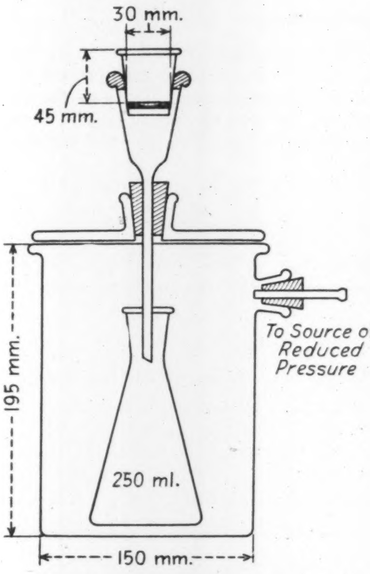

FIG. 1.—Filtration Apparatus.

(*b*) *Filter Aid.*—Hyflo[23] grade of diatomaceous earth, or equivalent.

(*c*) *Electric Oven.*—An electric oven capable of maintaining a temperature of 105 ± 2 C. (221 ± 3.6 F.).

(*d*) *Balances.*—An analytical balance sensitive to 0.1 mg. for weighing the Gooch crucible; a laboratory balance sensitive to 0.1 g. for weighing the oil sample.

(*e*) *Tamping Rod.*—A glass tamping rod having a flattened disk approximately 3 mm. in diameter at one end.

(*f*) *Flasks.*—Erlenmeyer flasks of 125- and 250-ml. capacity.

(*g*) *Desiccator.*

Reagents:

A.S.T.M. Precipitation Naphtha.—The A.S.T.M. precipitation naphtha shall conform to the requirements prescribed in Section A19 (*a*) (method A).

(*b*) *Chloroform, U.S.P.*

Procedure:

A25. (*a*) Heat the sample, while agitating constantly, to about 60 C. (140 F.) until the sample is homogeneous and all sediment is in suspension.

(*b*) Weigh 10 ± 0.1 g. of the prepared sample into a 125-ml. Erlenmeyer flask. Add 100 ± 1 ml. of the solvent and shake until the mixture is homogeneous. Stopper and let stand at room temperature for 1 to 2 hr.

(*c*) Add approximately 4 g. of filter aid to the clean Gooch crucible and dry in the oven at 105 ± 2 C. (221 ± 3.6 F.) to constant weight (30 to 60 min. is usually sufficient). Cool in a desiccator, and weigh the crucible and filter aid to the nearest 0.1 mg. Assemble the crucible and flask in the vacuum apparatus as indicated in Fig. 1 and reduce the pressure. The filter aid should be wet with the solvent from a wash bottle and the filter aid packed down with the glass rod. All filter aid should be washed from the rod into the crucible.

(*d*) Filter the contents of the Erlenmeyer flask through the wet filter bed without allowing the filter cake to suck dry (Note 1). All insoluble material should be transferred from the flask to the crucible by swirling the flask when adding solution to the crucible and by using increments of solvent to wash the flask. A total of 100 ± 1 ml. of fresh solvent shall then be used to wash the flask and filter cake. If, during filtration, the rate slows down because of a gummy deposit on the surface of the filter cake, the surface may be broken up with a pointed glass rod and the filtering continued (Notes 2 and 3). Material adhering to the glass rod should be washed with the solvent into the crucible.

NOTE 1.—During filtration, suction should be so controlled that no precipitate is drawn through the filter bed. As a rule the reduced pressure may be as low as 10 cm. of mercury. In some cases less reduction in pressure must be used to prevent loss of precipitate. If the filtrate is cloudy, it should be refiltered and the sum of the precipitates used in the calculations.

NOTE 2: *Modified Procedure.*—Some used lubricating oils will form naphtha insoluble matter of a gelatinous consistency which will not filter at a reasonable rate. In such cases the filtration may be accelerated by placing about half of the weighed dry filter aid in a new sample. This can be accomplished without loss of filter aid by use of a short-stemmed glass funnel inserted in the flask and gently tapping the crucible against the edge of the funnel. The adhering filter aid should then be washed from the funnel into the sample with the 100 ml. of solvent. During filtration the contents of the Erlenmeyer flask are swirled to distribute the filter aid evenly. After filtration, the filter aid clinging to the sides of the flask is transferred to the crucible by means of a jet of the solvent from the wash bottle, and the determination is completed in accordance with the regular procedure.

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[23] Available from Johns-Manville Co., Manville, N. J.

NOTE 3.—If the porous glass filter plate becomes clogged with fine particles such as soot from diesel oils, it may be cleaned by washing with nitric acid and then with chromic acid cleaning solution drawn through the plate in the reverse direction.

(*e*) After liquid solvent has been sucked from the washed filter cake, dry the crucible and contents in the oven for 3 to 4 hr. at 105 ± 2 C. (221 ± 3.6 F.), cool in a desiccator, and weigh to the nearest 0.1 mg.

Calculation:

A26. Calculate the percentage of insoluble matter as follows:

Insoluble matter, per cent = $10(B - A)$

where:

A = weight in grams of the prepared, dried Gooch crucible and filter aid, and

B = weight in grams of Gooch crucible and residue.

Reproducibility:

A27. With proper attention to details of preparation of the sample and procedure, the results of individual determinations should not differ from the average percentage of insoluble matter by more than 5 per cent of the average value.

APPENDIX VI

ENGINE TEST LOG SHEET

Test No.............................

Date.....................................

No. sheets...........................

Sheet No...

Brand name or description of test oil..

.... Viscosity grade...................................

Date	Time Start	Time Stop	Elapsed Time, hr.	Engine Speed, rpm.	Beam Load, lb.	Crank-case-oil Sump Temperature, deg. Fahr.	Oil Pressure, psi.	Water Temperature, deg. Fahr.		Intake Manifold Vacuum, inches of mercury	Exhaust Pressure, inches of mercury	Oil Added qt.		
								Inlet	Outlet					

Observers..

APPENDIX VII

SUGGESTED REPORT FORM

Test No..........
Date..........

GENERAL INFORMATION

Brand name or experimental designation of oil..........
.... Viscosity grade..........
Engine No.......... Year.......... Duration of test, hr..........
Crankcase-oil sump temperature, deg. Fahr.......... Load, hp.......... Speed, rpm..........
Total oil added,[a] qt.......... Total oil drained[b], qt.......... Quarts per hour..........
Coolant temperature, deg. Fahr., Inlet.......... Outlet.......... Spark plug gap, in..........
Cam angle, deg.......... or breaker-point gap, in.......... Start.......... Finish..........
Ignition advance, deg. b.t.d.c. at 3150 rpm.......... Start.......... Finish..........
Intake-valve clearance, in.......... Start.......... Finish..........
Exhaust-valve clearance, in.......... Start.......... Finish..........
Oil pressure, psi.......... Start.......... Finish..........
Intake-manifold vacuum, inches of mercury.......... Start.......... Finish..........
Exhaust back pressure, inches of mercury.......... Start.......... Finish..........
Fuel rate, time for consumption of 300 ml., min.......... Start.......... Finish..........
Exhaust gas:
Carbon dioxide, per cent.......... Start.......... Finish..........
Oxygen, per cent.......... Start.......... Finish..........
Carbon monoxide, per cent.......... Start.......... Finish..........
Air-fuel ratio.......... Start.......... Finish..........

VARNISH DEPOSIT RATINGS	Rating	SLUDGE DEPOSIT RATINGS	Rating
Piston skirts		Rocker-arm assembly	
Rocker-arm cover plate		Rocker-arm cover plate	
Push-rod cover plate		Push-rod cover plate	
Cylinder walls		Oil screen	
Crankcase oil pan		Crankcase oil pan	
Varnish total		Sludge total	

Combined engine-deposit rating for varnish and sludge..........

FUEL
Octane Number.......... Sulfur.......... Lead.......... ml. tetraethyl lead per gal.

COPPER-LEAD BEARINGS

Bearing number			
Clearance, in.			
Weight of bearing, g.:			
Top section	Start		
	Finish		
	Loss		
Bottom section	Start		
	Finish		
	Loss		

OIL ANALYSIS

Sample No.	Hours	Neutralization Number	Naphtha-Insoluble Matter, per cent	Naphtha-Insoluble Matter Soluble in Chloroform, Method A, per cent	Asphaltenes, Method B, per cent	Conradson Carbon Residue, per cent	Ash, per cent	Viscosity, Saybolt Universal, at 100 F., sec.

REMARKS..........
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[a] Including oil added to compensate for withdrawal of samples. See Section 12.
[b] Including volume of ½-pt. samples. See Section 12.

REPORT OF SECTIONAL COMMITTEE
ON
PETROLEUM PRODUCTS AND LUBRICANTS

ASA PROJECT: Z11

Sectional Committee Z11 on Petroleum Products and Lubricants held one meeting during the year in Chicago, Ill., on June 24, 1941.

During the past year, the American Standards Association, on the recommendation of the sectional committee, reapproved three American Standards in which revisions have been made, and likewise approved four A.S.T.M. standards as American Standard for the first time, assigning the following ASA designations:

A.S.T.M. Standard Methods of:

- Test for Carbon Residue of Petroleum Products (Conradson Carbon Residue) (D 189 - 41; ASA Z11.25-1941),
- Test for Distillation of Gas Oil and Similar Distillate Fuel Oils (D 158 - 41; Z11.26-1941),
- Test for Distillation of Crude Petroleum (D 285 - 41; Z11.32-1941),
- Test for Distillation of Plant Spray Oils (D 447 - 41; Z11.43-1941),
- Test for Vapor Pressure of Petroleum Products (Reid Method) (D 323 - 41; Z11.44-1941),
- Calculating Viscosity Index (D 567 - 41; Z11.45-1941), and
- Conversion of Kinematic Viscosity to Saybolt Universal Viscosity (D 446 - 39; Z11.46-1941).

Inasmuch as the A.S.T.M. Standard Method of Test for Knock Characteristics of Motor Fuels (D 357 - 40; ASA Z11.37-1940) has been revised and reverted to tentative, approval as American Standard has been withdrawn by the ASA on the recommendation of the sectional committee.

At its meeting held during this annual meeting, the following two tentative standards were recommended by Committee D-2 on Petroleum Products and Lubricants for reference to letter ballot of the Society for adoption as standard. The sectional committee decided to recommend to the American Standards Association the approval of these methods as American Standard upon their adoption as A.S.T.M. standards:

A.S.T.M. Tentative Methods of:

- Test for Carbon Residue of Petroleum Products (Ramsbottom Carbon Residue) (D 524 - 41 T), and
- Test for Tetraethyl Lead in Gasoline (D 526 - 41 T).

These will accordingly be referred to letter ballot in the sectional committee.

Since the standards listed below, already having ASA approval, have been revised this year, Committee D-2 has recommended that they be presented to the ASA in their revised form for reapproval as American Standard:

Revision of A.S.T.M. Standard Methods of:

- Test for Gum Content of Gasoline (D 381 - 36; ASA Z11.36-1936),
- Test for Flash Point by Means of the Pensky-Martens Closed Tester (D 93 - 40; Z11.7-1940),
- Test for Melting Point of Paraffin Wax (D 87 - 37; Z11.4-1937), and
- Test for Vapor Pressure of Petroleum Products (Reid Method) (D 323 - 41; Z11.44-1941.

The sectional committee voted to refer these revisions to the ASA, subject to approval by letter ballot.

The election of officers and other members of the Executive Committee of Sectional Committee Z11 for the ensuing term of two years resulted in the selection of the following:

Chairman, T. A. Boyd.
Vice-Chairman, T. G. Delbridge.

Other members of Executive Committee:

R. P. Anderson	J. B. Macauley
A. L. Beall	K. G. Mackenzie
H. C. Dickinson	J. A. Reid

Respectfully submitted on behalf of the sectional committee,

T. A. BOYD,
Chairman.

R. E. HESS,
Secretary.

REPORT OF COMMITTEE D-3
ON
GASEOUS FUELS

Committee D-3 on Gaseous Fuels held a joint meeting with its seven subgroups in Atlantic City, N. J., on October 19, 1941. Its Advisory Committee and the Subcommittee on Determination of Special Constituents of Gaseous Fuels also met on the same date.

Substantial progress has been made by various subcommittees in their assignments and several proposed tentative standards are expected to be completed within the next few months. Brief reports on the activities of these groups are presented below.

Subcommittee II on Measurement of Gaseous Samples (H. S. Bean, chairman).—Investigation of procedure for testing laboratory wet gas meters has progressed to the point where calibrations with the aspirator equipment and 0.1 cu. ft. bottle agree at the average rate at which calibration with the 0.1 cu. ft. bottle is made. However, these meters do not have a flat calibration curve, but tend to become "fast" as the rate increases. The rate of this change varies with different meters but the range of it is such that if a meter is correct at about 3 cu. ft. per hr. the average rate of a 0.1 cu. ft. bottle test, the meter may be from 0.1 to 0.3 per cent fast at 6 cu. ft. per hr., the rate of use in most calorimeter work.

A method of altering a wet test meter so as to obtain a flat calibration curve over a fairly wide range of rates, has been worked out. An application for a patent, to be dedicated to the public, is being made to cover this development.

Subcommittee III on Determination of Calorific Value of Gaseous Fuels (R. B. Harper, chairman).—Principal attention has been devoted to a study of routine determinations of calorific value of gaseous fuels by a special subgroup formed for the purpose. A proposed tentative method has been prepared and will shortly be submitted to the members for study and criticism.

An additional subgroup has been formed to study calorimeter thermometers in order to prepare suitable specifications for their manufacture.

Proposed definitions pertaining to calorific value of gaseous fuels prepared by a special subgroup were approved with minor revisions at the meeting of Committee D-3 held in Atlantic City on October 19, 1941. They have since been submitted to all members and approved by letter ballot.

Subcommittee IV on Determination of Specific Gravity and Density of Gaseous Fuels (E. F. Schmidt, chairman).—Test work was completed by the National Bureau of Standards on eleven specific gravity instruments submitted by their manufacturers. Fifteen test gases were employed and a total of more than 2000 specific gravity determinations were made during this study. Individual reports covering each instrument tested were prepared and a copy submitted to the manufacturer with the request that errors of fact be indicated so that suitable modifications could be made. After comments were received, a composite report was prepared and copies submitted to all members of the subcommittee as well as to all members of Committee D-3.

This report contains 239 pages of test

data and graphs. As soon as it has been fully reviewed by the subcommittee it is proposed to arrange for its release to industry through publication in the *Journal of Research* of the National Bureau of Standards.

Subcommittee V on Determination of Special Constituents of Gaseous Fuels (E. F. Pohlmann, chairman).—As the result of action taken by the Advisory Committee of Committee D-3 at its meeting on October 19, 1941, the former title of this subcommittee was changed. As a result, its objective is now the development of standard methods of procedure for the determination of special constituents of gaseous fuels.

This group has been devoting all of its efforts toward the preparation of standard methods for the determination of hydrogen sulfide, organic sulfur, and total sulfur.

Subcommittee VI on Determination of Water Vapor Content of Gaseous Fuels (A. W. Gauger, chairman).—Experimental work has been continued on methods of measuring the water content of gaseous fuels. An improved portable colorimeter employing the change in color of cobaltous bromide with water was constructed and given a field test. The moisture content indicated by the cobaltous bromide method was checked against the indication of a dew-point meter. Since most samples contained some "entrained" water, the cobaltous bromide method invariably indicated a higher water content; for example, the water content of the gas leaving an experimental diethylene glycol dehydrator was over twice that indicated by the dew-point reading at the same sampling location. Field tests indicate the need of a better sampling technique and this is being developed.

The infrared spectrometer is being calibrated. The amount of water vapor in a vacuum is readily measured to plus or minus 0.1 mm. of water vapor pressure and the absorption obeys the Lambert-Beer law. The effect of the partial pressure of other gases, and the temperature of the mixture affects the amount of light absorbed by the water vapor in the 1.8μ band. Although the spectrometer can still be used for quantitative measurements, the magnitude of this extra effect requires that the spectrometer be calibrated with known amounts of water. Consequently, the method cannot be the semiabsolute method anticipated.

Subcommittee VII on Complete Analysis or Chemical Composition of Gaseous Fuels (Martin Shepherd, chairman).—Results of the cooperative analysis of the first standard sample of carburetted water-gas type were completed and presented to the committee as tabular data and as frequency distribution plots. These data gave the first reasonably complete and authentic picture of the actual state of this type of gas analysis in this country at this time.

The second standard sample of the natural gas type for analysis by absorption and combustion was distributed May 5, 1941, and analyzed by 32 laboratories during the year. A complete report now is in preparation.

Cylinders for a third sample of the natural gas type for analysis by fractional distillation have been purchased. When the cylinders have been equipped with valves, the sample will be prepared and distributed.

This report has been submitted to letter ballot of the committee which consists of 33 members, 28 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

A. W. GAUGER,
Chairman.

R. M. CONNER,
Secretary.

REPORT OF COMMITTEE D-4

ON

ROAD AND PAVING MATERIALS

Committee D-4 on Road and Paving Materials has held two meetings during the past year: in Chicago, Ill., on June 25, 1941, and in Cleveland, Ohio, on March 4, 1942.

Subsequent to the 1941 annual meeting Committee D-4 presented to the Society, through Committee E-10 on Standards, the following recommendations:

New Tentative Standards:

Specifications for Sodium Chloride (D 632 – 41 T),
Specifications for Slow-Setting Emulsified Asphalt (for Fine Aggregate Mixes) (D 631 – 41 T), and
Volume Correction Table for Tar and Coal-Tar Pitch (D 633 – 41 T), submitted jointly with Committee D-8 on Bituminous Waterproofing and Roofing Materials.

Tentative Revisions of Standards:

Methods of Chemical Analysis of Calcium Chloride (D 345 – 34), and
Method of Test for Ductility of Bituminous Materials (D 113 – 39).

Revisions of Tentative Standards:

Specifications for Standard Sizes of Coarse Aggregate for Highway Construction (D 448 – 37 T),
Specifications for Crushed Slag for Bituminous Macadam Base and Surface Courses (D 487 – 39 T),
Specifications for Crushed Stone for Bituminous Macadam Base and Surface Courses (D 192 – 39 T), and
Methods of Sampling Bituminous Materials (D 140 – 39 T).

These recommendations were accepted[1] by Committee E-10 on August 25, 1941, and the new and revised tentative standards and tentative revisions of standards appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part II.

Subsequent to the 1941 annual meeting Committee D-4 also referred to Committee E-8 on Nomenclature and Definitions a proposed tentative revision of Standard Definitions of Terms Relating to Materials for Roads and Pavements (D 8 – 33) including definitions for the terms bituminous emulsions, fluxes, asphalts, rock asphalt, petroleum, topped petroleum, tars, water-gas tars, dehydrated tars, dead oils, viscosity, matrix, petroleum flux, and bituminous.

I. REVISION OF STANDARDS, IMMEDIATE ADOPTION

Standard Method of Test for Determination of Bitumen (**D 4 - 27**)[2] *and Standard Method of Test for Proportion of Bitumen Soluble in Carbon Tetrachloride* (**D 165 - 27**).[2]—In order to describe more definitely the procedures and to include certain editorial improvements in the descriptions of these tests, revisions of these methods have been prepared and are recommended for immediate adoption. The revised methods are appended hereto[3] and the com-

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee D-4 reported the following results of the letter ballot vote from a committee membership of 133: D 632, D 631, and D 345, affirmative 73, negative 0, ballots marked "not voting" 9; D 113, affirmative 80, negative 1, ballots marked "not voting" 4; D 448, affirmative 76, negative 1, ballots marked "not voting" 6; D 487, affirmative 68, negative 1, ballots marked "not voting" 13; D 192, affirmative 75, negative 1, ballots marked "not voting" 4; D 140, affirmative 82, negative 0, ballots marked "not voting" 3; D 633, affirmative 69, negative 0, ballots marked "not voting" 10.

[2] 1939 Book of A.S.T.M. Standards, Part II, pp. 385, 388.

[3] These revised methods were adopted as standard by the Society and appear in their latest revised form in the 1942 Book of A.S.T.M. Standards, Part II, pp. 453 and 456, respectively.

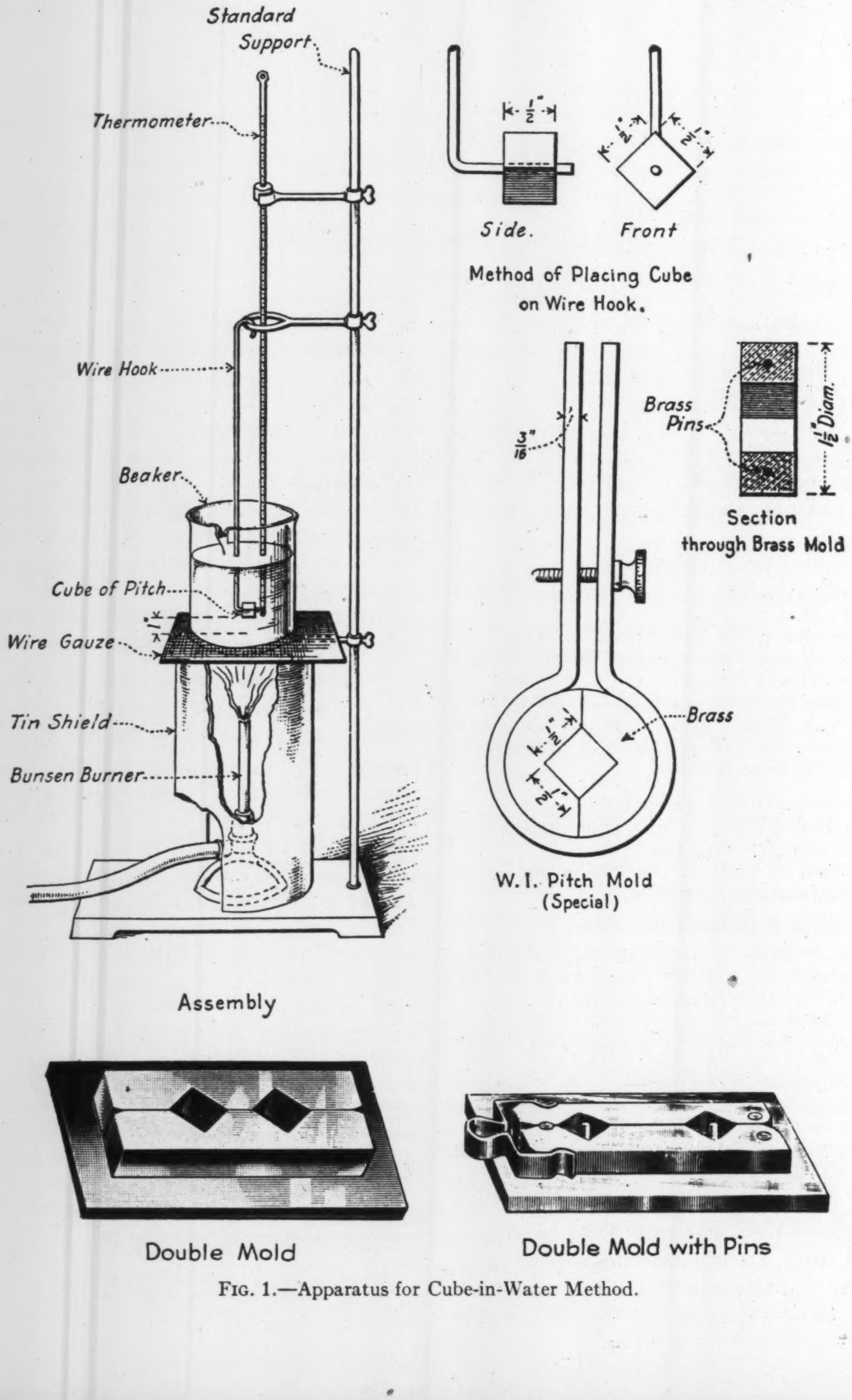
Standard Support
Thermometer
Wire Hook
Beaker
Cube of Pitch
1/2"
Wire Gauze
Tin Shield
Bunsen Burner
Assembly
1/2"
Side.
Front
Method of Placing Cube on Wire Hook.
3/16"
Brass Pins
1 1/2" Diam.
Section through Brass Mold
Brass
W. I. Pitch Mold (Special)
Double Mold
Double Mold with Pins

FIG. 1.—Apparatus for Cube-in-Water Method.

mittee accordingly requests a favorable nine-tenths vote at the annual meeting in order that these recommendations may be referred to letter ballot of the Society.

II. REVISION OF TENTATIVE STANDARD

Tentative Specifications for Crushed Slag for Bituminous Macadam Base and Surface Courses (**D 487 - 41 T**).[4]—The committee is proposing the following revisions in these tentative specifications and recommends that as revised they be continued as tentative:[5]

Section 2.—Change to read as follows by the addition of the italicized word and the omission of those in brackets:

> 2. The crushed slag shall be air-cooled blast-furnace slag and shall consist of angular fragments reasonably uniform in density and quality, and reasonably free from thin *or* elongated [or glassy] pieces, dirt, or other objectionable matter.

Section 3.—Insert the following requirements after the requirements for wear.

Weight per cubic foot, compacted, of each size specified:

Slag for use in base courses	not less than 65 lb.
Slag for use in surface courses	not less than 70 lb.

Section 6.—Add the following new Paragraph (*c*) after (*b*), relettering Paragraph (*c*) as (*d*):

> (*c*) *Weight per Cubic Foot.*—Standard Method of Test for Unit Weight of Aggregate (A.S.T.M. Designation: C 29).

III. ADOPTION AS STANDARD OF TENTATIVE REVISION OF STANDARD

Tentative Method of Test for Modified Miscibility and Cement Mixing of Emulsified Asphalts (**D 244 - 41 T**).[6]—Committee D-4 recommends that this method revised as indicated below be adopted as standard and incorporated in the Standard Methods of Testing Emulsified Asphalts (D 244 - 40):[7]

Section 6.—Change the second sentence to read as follows by the addition of the italicized word and figure and the omission of those in brackets:

> This test is not applicable [where 5] *when 2* per cent or more coagulation is anticipated.

Section 9 (d).—Change the first sentence to read as follows by the addition of the italicized words and figures and the omission of the words in brackets:

> Pour the mixture through the tared 1410-micron (No. 14) iron [wire] sieve *of approximately 3 in. diameter* and rinse [with water until the wash water is clear] *by pouring distilled water from a receptacle held at a height of approximately 6 in.*

IV. REINSTATEMENT OF TENTATIVE STANDARD

Upon recommendation of Committee D-4 in 1940 the Society discontinued the Tentative Method of Test for Abrasion of Gravel by Use of the Deval Machine (D 289 - 37 T). The recommendation was originally made because of the general rapid substitution of the Los Angeles machine test for the Deval machine test. However, a number of the state highway departments are continuing to use the Deval machine and it is still a standard method of the American Association of State Highway Officials. Moreover, in 1940 the Deval machine test was included in specifications of the Joint Committee on Concrete and Reinforced Concrete. For these reasons Committee D-4 feels that it is advisable to reinstate this method of test as an A.S.T.M. tentative standard and so recommends to the Society.

V. EDITORIAL CHANGES IN STANDARDS AND TENTATIVE STANDARDS

Standard Method of Test for Softening Point of Tar Products (Cube-in-Water

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[4] 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 334.

[5] See also Editorial Note, p. 350.

[6] 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 347.

[7] 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 94.

Method) (**D 61 - 38**).[8]—Committee D-4 recommends that the following changes be incorporated in this standard and accepted as an editorial revision. These changes merely provide for use of a double mold if desired, in addition to the single mold and necessitate a modification in Fig. 1.

Section 2 (*a*).—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

> 2. (*a*) *Mold.*—A mold suitable for forming a 12.7-mm. (½-in.) cube of pitch. (*Suitable types are* [A recommended type is] shown in Fig. 1.)

Fig. 1.—Replace this figure by the accompanying Fig. 1.

Standard Method of Test for Softening Point of Bituminous Materials (*Ring-and-Ball Method*) (**D 36 - 26**).[9]—Committee D-4 recommends that the following correction be incorporated in this standard and be accepted as an editorial change. This change eliminates a contradiction in description of thermometer and is in conformity with the thermometer requirements of Committee E-1 on Methods of Testing.

Section 8.—Change to read as follows by the addition of the italicized figures and the omission of those in brackets:

> 8. An A.S.T.M. High Softening Point Thermometer, graduated in either Centigrade or Fahrenheit degrees as specified, having a range of 30 to *200* [160] C. or 85 to *392* [320] F. and conforming to the requirements for this thermometer as prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1) of the American Society for Testing Materials shall be used.

Tentative Specifications for Materials for Stabilized Base Course (**D 556 - 40 T**)[10] *and Tentative Specifications for Materials for Stabilized Surface Course* (**D 557 - 40 T**).[11]—Committee D-4 recommends that the following change be incorporated in both of these standards and accepted as editorial. This change is consistent with the sieve analysis requirements in Section 3 in which no reference is made to the No. 4 sieve, and the No. 10 sieve is used as the division point for Type A material.

Section 2 (*a*).—Change the second sentence to read as follows by the addition of the italicized figures and the omission of those in brackets:

> The aggregate retained on the No. *10* [4] (*2000*–[4760] micron) sieve shall consist of hard, durable particles and shall be free from injurious or deleterious substances.

Standard Methods of Testing Emulsified Asphalts (**D 244 - 40**).[7]—Committee D-4 recommends that in the interest of clarity the following change be incorporated in this standard and accepted as an editorial revision.

Section 21.—Change the second sentence to read as follows by the addition of the italicized words and the omission of those in brackets:

> The percentage of residue *retained on the sieve* [in the emulsion] shall be calculated *from* [on the basis of] this weight.

Standard Method of Test for Distillation of Tar Products (**D 20 - 30**).—Committee D-4 recommends that, in the interest of clarity and in order to harmonize this method with the revised Standard Method of Test for Distillation of Creosote (D 246 – 39) as recommended by Committee D-7 on Timber, the following changes be incorporated in this standard and accepted as an editorial revision.

Section 4 (*b*).—Change to read as follows by the addition of the italicized words:

> (*b*) The axis *of the bulb* of the flask through *the center* of the neck shall be vertical *and the thermometer shall be aligned on this axis.*

Section 4 (*c*).—Change the first sentence to read as follows by the addition of the italicized words and the omission of those in brackets:

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[8] 1939 Book of A.S.T.M. Standards, Part II, p. 420.

[9] *Ibid.*, p. 417.

[10] 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 272.

[11] *Ibid.*, p. 274.

The distance from the [bulb of the thermometer] *neck of the flask* to the outlet end of the condenser tube shall be not more than 600 nor less than 500 mm.

Fig. 3.—Change the 500–600 mm. dimension to conform with the above revision in Section 4 (*c*) and as shown in Fig. 3 of the Standard Method of Test for Distillation of Creosote (D 246 – 39) but without change in position to the thermometer.

VI. STANDARDS AND TENTATIVE STANDARDS CONTINUED WITHOUT REVISION

In order to determine whether they are in accord with present practice the committee has reviewed the following specifications and methods of test which have been published without revision for a number of years, and has approved retaining these standards and tentative standards in their present form:

Standard Specifications for:

Gravel for Bituminous Concrete Base (D 309 – 30),[12]
Sand for Sheet Asphalt and Bituminous Concrete Pavements (D 162 – 29),
Calcium Chloride (D 98 – 34),
Materials for Cement Grout Filler for Brick and Stone Block Pavements (D 57 – 20),
Coal-Tar Pitch for Stone Block Filler (D 112 – 30),

Standard Methods of Test for:

Toughness of Rock (D 3 – 18),
Abrasion of Rock by Use of the Deval Machine (D 2 – 33),
Chemical Analysis of Calcium Chloride (D 345 – 34),[13]
Distillation of Tar Products Suitable for Road Treatment (D 20 – 30),
Float Test for Bituminous Materials (D 139 – 27),
Penetration of Bituminous Materials (D 5 – 25),
Residue of Specified Penetration (D 243 – 36),[14]
Specific Gravity of Asphalts and Tar Pitches Sufficiently Solid to Be Handled in Fragments (D 71 – 27),
Specific Gravity of Road Oils, Road Tars, Asphalt Cements, and Soft Tar Pitches (D 70 – 27),

Tentative Specifications for:

Cut-Back Asphalt (Medium Curing Type) (D 598 – 40 T),
Cut-Back Asphalt (Rapid Curing Type) (D 597 – 40 T),
Tar (D 490 – 38 T),
Asphalt Filler for Brick Pavements (D 241 – 39 T),

Tentative Method of:

Test for Loss on Heating of Oil and Asphaltic Compounds (D 6 – 39 T). (This method of test is now in process of revision.),

Tentative Revision of:

Standard Method of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (C 131 – 39) issued June, 1941.

The following two new specifications and proposed revisions of six tentative standards have been referred to letter ballot of Committee D-4 for recommendation to the Society subsequent to the annual meeting through Committee E-10 on Standards.[12]

Proposed Tentative Standards:

Spec. for Crushed Gravel for Waterbound Base and Surface Courses
Spec. for Gravel for Bituminous Concrete Base and Surface Courses

Revisions of Tentative Specifications for:

Standard Sizes of Coarse Aggregate for Highway Construction (D 448 – 41 T)
Crushed Slag for Bituminous Concrete Base and Surface Courses (D 485 – 38 T)
Crushed Stone for Bituminous Concrete Base and Surface Courses (D 486 – 39 T)
Crushed Slag for Water-Bound Base and Surface Courses (D 488 – 38 T)
Crushed Stone for Water-Bound Base and Surface Courses (D 489 – 40 T)

Revision of Tentative Method of:

Sampling Stone, Slag, Gravel, Sand, and Stone Block for Use as Highway Materials (D 75 – 40 T)

In connection with the new and revised specifications for stone, slag, and gravel, it is proposed to consolidate these into three separate specifications.[12]

In addition Committee D-4 has referred to letter ballot the Proposed Method of Test for Moisture-Density Relations of Soils, originated by Committee D-18 on Soils for Engineering

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[12] See Editorial Note, p. 350.

[13] Tentative revision of this standard issued August, 1941.

[14] These specifications are now in process of being revised.

Purposes but coming under the joint jurisdiction of the two committees.[12]

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 129 members; 89 members returned their ballots with the results shown in Table I.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, C. N. Forrest.
First Vice-Chairman, F. V. Reagel.
Second Vice-Chairman, A. T. Goldbeck.
Third Vice-Chairman, E. O. Rhodes.
Secretary, Prévost Hubbard.

This report has been submitted to letter ballot of the committee which consists of 129 members; 89 members returned their ballots, of whom 78 have voted affirmatively and 1 negatively.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. REVISION OF STANDARDS, IMMEDIATE ADOPTION			
Test for Determination of Bitumen (D 4 – 27; ASA A37.3-1930)	73	0	16
Test for Proportion of Bitumen Soluble in Carbon Tetrachloride (D 165 – 27)	73	0	16
II. REVISION OF TENTATIVE STANDARD			
Spec. for Crushed Slag for Bituminous Macadam Base and Surface Courses (D 487 – 41 T)	59	0	30
III. ADOPTION AS STANDARD OF TENTATIVE REVISION OF STANDARD			
Testing Emulsified Asphalts (D 244 – 40)	55	6	21
IV. REINSTATEMENT OF TENTATIVE STANDARD			
Test for Abrasion of Gravel by Use of the Deval Machine (D 289 – 37 T)	61	3	25

Respectfully submitted on behalf of the committee,

J. E. MYERS,
Chairman.

PRÉVOST HUBBARD,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee D-4 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative Specifications for:

Crushed Stone, Slag, and Gravel for Bituminous Concrete Base and Surface Courses of Pavements (D 692 – 42 T), to replace immediately Specifications D 485 – 38 T, D 486 – 39 T, and D 309 – 30,

Crushed Stone and Crushed Slag for Bituminous Macadam Base and Surface Courses of Pavements (D 693 – 42 T), to replace immediately Specifications D 192 – 41 T and D 487 – 42 T,

Crushed Stone, Slag, and Gravel for Water-Bound Base and Surface Courses of Pavements (D 694 – 42 T), to replace immediately Specifications D 488 – 38 T and D 489 – 40 T,

Revision of Tentative Specifications for:

Standard Sizes of Coarse Aggregate for Highway Construction (D 448 – 41 T), and

Revision of Tentative Methods of:

Sampling Stone, Slag, Gravel, Sand, and Stone Bock for Use as Highway Materials (D 75 – 40 T).

These recommendations were accepted by Committee E-10 on August 24, 1942, and the new and revised tentative standards appear in the 1942 Book of A.S.T.M. Standards, Part II, pp. 1224, 1230, 1218, and 1233.

The proposed Method of Test for Moisture-Density Relations of Soils, submitted jointly by Committees D-4 and D-18, were accepted by Committee E-10 on September 17, 1942, for publication as tentative. This new tentative method was assigned the designation D 698 – 42 T and appears in the 1942 Book of A.S.T.M. Standards, Part II, p. 1280.

REPORT OF COMMITTEE D-5

ON

COAL AND COKE

Committee D-5 on Coal and Coke held a meeting in Chicago, Ill., on June 25, 1941; another meeting will be held at the annual meeting of the Society in June, 1942, in Atlantic City, N. J. Three of its subcommittees met in Easton, Pa., on October 10, 1941, in connection with a joint meeting of the Fuels Division of the American Society of Mechanical Engineers, and the Coal Division of the American Institute of Mining and Metallurgical Engineers.

A new subcommittee designated as Subcommittee XIX on Designation of Coal Sizes, under the chairmanship of J. D. Doherty, consisting of 11 members, has been formed to consider revising the Standard Method for Designating the Size of Coal from Its Screen Analysis (D 431 – 38). This subcommittee also will have jurisdiction over the Standard Method of Test for Screen Analysis of Coal (D 410 – 38) and over all matters concerning coal size designations that may come up in the future.

In accordance with the Regulations Governing Standing Committees reference is made here to standards that have stood for six years without revision and tentative standards that have stood for two years without revision.

Revisions are in preparation for the following tentative standards which have stood for two years or more without revision:

Tentative Methods of:

Sampling Coals Classed According to Ash Content (D 492 – 40 T),

Test for Grindability of Coal by the Ball-Mill Method (D 408 – 37 T),

Test for Grindability of Coal by the Hardgrove-Machine Method (D 409 – 37 T),

Drop Shatter Test for Coal (D 440 – 37 T),

Tumbler Test for Coal (D 441 – 37 T), and

Tentative Definition of Terms Gross Calorific Value and Net Calorific Value of Fuels (D 407 – 35 T).

Standards that have stood for six years without revision and on which no revisions are in progress are as follows:

Specifications for Gas and Coking Coals (D 166 – 24),

Method of Sampling and Fineness Test of Powdered Coal (D 197 – 30),

Test for Sieve Analysis of Crushed Bituminous Coal (D 311 – 30),

Test for Cubic Foot Weight of Crushed Bituminous Coal (D 291 – 29),

Definitions of Terms Relating to Coal and Coke (D 121 – 30),

Method of Sampling Coke for Analysis (D 346 – 35),

Test for Volume of Cell Space of Lump Coke (D 167 – 24),

Method of Drop Shatter Test for Coke (D 141 – 23),

Method of Tumbler Test for Coke (D 294 – 29),

Test for Sieve Analysis of Coke (D 293 – 29), and

Test for Cubic Foot Weight of Coke (D 292 – 29).

Under consideration is a revision of the Standard Method of Test for Size of Anthracite (D 310 – 34) which will change the requirements for the screens for testing egg-size anthracite to conform to the current specifications of the Anthracite Committee, Pennsylvania Department of Commerce, which committee now has jurisdiction over the anthracite sizing specifications formerly under the Anthracite Institute.

ADOPTION AS STANDARD OF TENTATIVE REVISION OF STANDARD

Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 – 40).[1]—The committee recommends that the tentative revision in Section 43, in this standard method, as published in 1940,[2] pertaining to tolerances for volatile matter determination be approved for reference to letter ballot of the Society for adoption as standard. The revisions of Sections 27 to 31 which pertain to the ash-fusion test are to remain as tentative.

This recommendation has been submitted to letter ballot of the committee which consists of 53 members; 40 members returned their ballots of whom 38 have voted affirmatively, 1 negatively, and 1 marked his ballot "not voting."

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Methods of Testing (W. A. Selvig, chairman).—A Special Section on Volumetric Methods for Sulfur Determination, under the chairmanship of C. A. Lunn, consisting of 9 members, has completed a comprehensive study of volumetric methods for determination of sulfur in coal and coke. This investigation was under the immediate supervision of S. S. Tomkins.

Samples of four coals ranging in sulfur content from approximately 0.6 to 3.3 per cent were air-dried, reduced and ground to pass through a 250-micron (No. 60) sieve and representative portions submitted to eight laboratories. These laboratories determined sulfur in triplicate by the present standard Eschka and bomb-washing methods whereby the sulfur is determined gravimetrically by weighing as barium sulfate, and by two volumetric methods using tetrahydroxyquinone and sodium rhodizonate as indicators. In these volumetric methods sulfur is recovered as soluble sulfates in the bomb washings or by leaching the Eschka residues. The advantages claimed for the volumetric methods are that they are less costly and time-consuming, especially where a large number of coal samples are tested daily.

On the basis of the results reported by the cooperating laboratories it was concluded that the volumetric methods will yield results of the same degree of reproducibility and accuracy as the present standard gravimetric method. The two volumetric methods are now under consideration as alternatives to the present standard gravimetric method. The detailed investigation is given in a paper entitled "Volumetric Determination of Sulfur in Coal and Coke," by S. S. Tomkins, *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 14, pp. 141 to 145 (1942).

A Special Section on Uniformity of Analytical Data, under the chairmanship of H. H. Lowry, has completed a study of application of statistical methods to determine the precision of the procedures given for analysis of coal and coke in Standard Methods D 271. A paper covering this investigation entitled "Statistical Study of the Precision of Methods for Analysis of Coal and Coke,"[3] by H. H. Lowry and Charles O. Junge, Jr., of the Coal Research Laboratory, Carnegie Institute of Technology, is to be presented at this annual meeting of the Society. A knowledge of this precision is of particular importance in any attempt to correlate performance of coals in various uses with coal analyses.

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[1] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 5.

[2] *Ibid.*, p. 561.

[3] See p. 870.

Nineteen Governmental, institutional, industrial, and commercial laboratories collaborated in this study by furnishing the analytical data in duplicate, or multiplicate, generally on 100 samples of coal or coke. In order to avoid bias in the selection of the data reported, each laboratory was asked to report the data beginning with the most recent determination of any given quality and continuing in inverse chronological order, with no omission, the results of individual analyses up to 100 samples. These data were then analyzed by the method described by Deming and Birge in the *Review of Modern Physics*, Vol. 6, pp. 158 to 160 (1934). This method of analysis assumes a distribution of errors in accord with the normal law of errors. Study of the data submitted has shown that this assumption may not be justified for only a few sets of data. There are presented in the paper the numerical results of the statistical study which should permit establishment of "permissible differences" of duplicate determinations on a single sample of coal or coke related definitely to the precision of the analytical procedures. The results considered in the paper do not permit any estimate to be made of the absolute accuracy of the analytical procedures covered in Standard Methods D 271, although similar statistical methods could be applied to data if they can be made available.

To obtain information as to how best to specify the nature of the reducing atmosphere in the standard coal-ash fusion test a cooperative investigation has been completed in which seventeen different laboratories took part. Gas-fired and electrically heated furnaces were used. The fuels used for heating the gas-fired furnaces included natural gas, manufactured gas, coke-oven gas, and oxygen-acetylene mixtures. The cooperating laboratories determined in duplicate the initial deformation temperature, the softening temperature, and the fluid temperature of a series of coal ashes covering a wide range of fusibility. The data reported are being tabulated for publication as a Bureau of Mines Report of Investigations.

The subcommittee has recommended the adoption as standard of the tentative revision[2] of Section 43 of the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 - 40) which pertains to tolerances for the volatile matter determination. All members of the subcommittee returned their ballots and the vote was 12 affirmative with 1 ballot marked "not voting."

Subcommittee VII on Pulverizing Characteristics of Coal (John Van Brunt, chairman).—This subcommittee held a meeting in Easton, Pa., on October 10, 1941. Considerable experimental work has been done on the Lea-Nurse air permeability method for measuring specific surfaces of finely ground coal and the application of this procedure to the present Tentative Method of Test for Grindability of Coal by the Hardgrove-Machine Method (D 409 - 37 T). At the 1941 annual meeting of the Society a paper[4] was presented covering such an investigation conducted in the laboratories of the Babcock & Wilcox Co. at Barberton, Ohio.

Experimental work also has been conducted at the Battelle Memorial Institute at Columbus, Ohio, concerning the application of the Lea-Nurse air-permeability method to the ball-mill method for determination of coal grindability (D 408 - 37 T) and the results of this investigation[5] have been made available to members of the subcommittee.

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[4] J. B. Romer, "A Study of the Grindability of Coal and the Fineness of Pulverized Coal When Using the Lea-Nurse Air Permeability Method for Evaluating the Subsieve Fractions," *Proceedings*, Am. Soc. Testing Mats., Vol. 41, pp. 1152 to 1166 (1941).

[5] Ralph A. Sherman, and George W. Land, "The Determination of the Grindability of Coal by Use of the Ball-Mill and the Lea-Nurse Air-Permeability Apparatus," Report in mimeographed form circulated to members of Subcommittee VII, of A.S.T.M. Committee D-5.

The subcommittee plans on continuing its investigations of the Lea-Nurse method for determining specific surfaces of pulverized coal and its application to the present two tentative methods for coal grindability.

Subcommittee XIII on Coal Sampling (Henry F. Hebley, chairman).—This subcommittee held a meeting in Easton, Pa., on October 10, 1941. It intends to do some experimental work relative to the number of increments required in collecting samples of coal with top size ranging from 4 to 6 in. The principal activity of the subcommittee this last year has been in connection with combining the Standard Method of Sampling Coal for Analysis (D 21 - 40) with the Tentative Method of Sampling Coals Classed According to Ash Content (D 492 - 40 T) and considerable progress has been made in this proposed consolidation. The work on methods of sampling coal for determination of moisture has been recessed temporarily.

Subcommittee XV on Plasticity and Swelling of Coal (C. C. Russel, chairman).—In connection with the work of this subcommittee a paper entitled "An Experimental Investigation of the British Standard Method for the Crucible Swelling Test for Coal," by H. N. Ostborg, H. R. Limbacher, and Ralph A. Sherman is to be presented at this annual meeting of the Society.[6] The paper describes experiments in standardizing a method for evaluating free swelling characteristics of coals when burned in fuel beds.

Subcommittee XIX on Designation of Coal Sizes (J. D. Doherty, chairman).—This subcommittee held an organization meeting in Easton, Pa., on October 29, 1941. A survey is being made of current usage in expressing sizes of coal. The present Standard Method for Designating Size of Coal from its Screen Analysis (D 431 - 38) gives the lower limiting screen first followed by the upper limiting screen, as for example 2 to 4 in. In some quarters it is customary to give the upper limiting screen first. After the survey of current practice in expressing coal sizes has been completed the subcommittee will consider revisions of the present standard that might be desirable.

The present officers of the committee were re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 53 members; 40 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

A. C. FIELDNER,
Chairman.

W. A. SELVIG,
Secretary.

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[6] See p. 851

REPORT OF COMMITTEE D-6
ON
PAPER AND PAPER PRODUCTS

Committee D-6 on Paper and Paper Products held one meeting during the year, on June 25, 1941, in Chicago, Ill.

Cooperative relations are being continued between Committee D-6 and other A.S.T.M. committees as indicated in the following table:

A.S.T.M. Committee	D-6 Representative on Committee	Representative on Committee D-6
C-16 on Thermal Insulating Materials	C. C. Heritage	....
D-8 on Bituminous Waterproofing and Roofing Materials	....	M. R. Beasley
D-9 on Electrical Insulating Materials	J. M. Finch	R. W. Chadbourne
Joint D-6 and D-9 Committee on pH of Paper	Helen Kiely M. S. Kantrowitz J. M. Finch	B. L. Clark M. Guarnier C. F. Hanson
D-10 on Shipping Containers	....	Edward Dahill
Committee E-1 on Methods of Testing:		
Tech. Comm. III on Particle Size and Shape	W. R. Willets	....
Tech. Comm. X on Conditioning and Weathering	L. S. Reid	....
Tech. Comm. XIII on Hydrogen Ion Determinations	M. S. Kantrowitz	....
Joint A.S.T.M.-TAPPI Committee on Paper Testing Methods	G. H. Harnden R. C. Griffin	P. F. Wehmer B. L. Wehmhoff

The following additions in membership have been approved during the year:

M. R. Beasley, Bird and Son, Inc. (Representing Committee D-8)

W. E. Harvey, Irvington Varnish and Insulator Co.,

P. K. Baird, U. S. Forest Products Laboratory,

T. A. McElhanney, Canada Forest Products Laboratories,

Melbourne Heinig, U. S. Forest Products Laboratory,

C. L. Sheldon, Hood Rubber Co., as representative of the National Assn. of Purchasing Agents, replacing J. M. Berry who has been called to active duty in the Ordnance Dept. of the U. S. Army; and

R. G. Betts, as representative of W. & L. E. Gurley, replacing W. L. Egy who is no longer with that organization.

W. E. Emley resigned as chairman of Subcommittee I on Paper Test Methods, and W. R. Willets was appointed as the new chairman of this subcommittee.

At the present time, the committee consists of 66 members, of whom 54 are voting members, 24 are classified as producers, 20 as consumers, 17 as general interest members, and 5 as consulting members.

Subsequent to the 1941 annual meeting, Committee D-6 presented to the Society through Committee E-10 on Standards the following new tentative standards:

New Tentative Methods of:

Conditioning of Paperboard, Fiberboard and Paperboard Containers for Testing (D 641 - 41 T),

Compression Testing of Corrugated and Solid Fiber Boxes (D 642 - 41 T),

Test for Folding Endurance of Paper (D 643 - 41 T),

Test for Moisture in Paper and Paper Products (D 644 - 41 T),

Test for Thickness of Paper (D 645 - 41 T), and

Test for Basis Weight of Paper and Paper Products (D 646 - 41 T).

These tentative methods were accepted[1] by Committee E-10 on August 25, 1941, and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part III.

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee D-6 reported the following results of the letter ballot vote of a total of 42 ballots returned from a membership of 56: D 641, affirmative 35, negative 3, ballots marked "not voting" 4; D 642, affirmative 36, negative 0, ballots marked "not voting" 6; D 643, affirmative 33, negative 0, ballots marked "not voting" 9; D 644, affirmative 35, negative 1, ballots marked "not voting" 6; D 645, affirmative 35, negative 0, ballots marked "not voting" 7; D 646, affirmative 34, negative 0, ballots marked "not voting" 8.

I. NEW TENTATIVE STANDARDS

The committee recommends that the following new methods be accepted for publication as tentative, as appended hereto:[2]

Tentative Methods of Test for:

Pentosans in Paper,
Conditioning Paper and Paper Products for Testing,
Quantitative Determination of Coating on Mineral-Coated Paper,
Internal Tearing Resistance of Paper, and
Qualitative Examination of Mineral Fillers and Mineral Coatings of Paper.

II. REVISIONS OF TENTATIVE STANDARDS

The committee recommends revisions in two tentative standards as given in detail below:

Tentative Method of Test for Basis Weight of Paper and Paper Products **(D 646 – 41 T)**:[3]

Section 9.—Change to read as follows by the addition of the italicized words and the omission of the words in brackets:

9. Duplicate determinations made on [different sets of] *the same* sample[s from the same shipment and on different apparatus] shall agree within 2 per cent.

Tentative Method of Test for Moisture in Paper **(D 644 – 41 T)**:[4] The following minor changes are recommended in this method so that it will also be applicable to testing paperboard and fiberboard containers. The inclusion of these changes in the method will relieve Subcommittee IV on Paperboard and Fiber Containers from preparing a separate method for the determination of moisture for paperboard and fiberboard containers.

Title.—Change the title of this method by the addition of the words, "Paperboard, and Paperboard and Fiberboard Containers."

Section 1.—Change to read as follows by the addition of the italicized words:

1. This method covers the procedure for determining moisture in all papers, *paperboards, fiberboard and paperboard containers,* except those containing matter other than water volatile at 100 to 105 C.

Section 2 (a).—Change to read as follows by the addition of the italicized words and the omission of the words in brackets:

(*a*) *Weighing Container.*—Either a wide-mouth, glass-stoppered bottle or an airtight metal container, *or any other suitable airtight container,* depending [up]on the size of the [specimen] *sample required.*

Section 3.—Add a new Paragraph (*c*) to read as follows:

(*c*) When the moisture is determined on combined board or containers which are to be tested for other physical properties, samples shall weigh approximately 50 g. and shall be taken so as to be representative of the material being tested. Samples shall be cut from unsealed sections and preferably from unprinted sections of the container, and shall be taken from each type of container being tested.

Section 5.—Reletter Paragraph (*b*) as (*c*) and add a new Paragraph (*b*) to read as follows:

(*b*) When the percentage of moisture based on bone-dry weight is required, it shall be calculated as follows:

$$\text{Moisture, per cent} = \frac{W_1 - W_2}{W_2} \times 100$$

where:
W_1 = weight of original sample, and
W_2 = weight of sample after drying.

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[2] These methods were accepted as tentative by the Society and appear in the 1942 Book of A.S.T.M. Standards, Part III, pp. 1022, 1013, 1020, 1025, 1016, respectively.

[3] 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 599.

[4] *Ibid.*, p. 594.

III. ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the following six tentative standards be approved without change for reference to letter ballot of the Society for adoption as standard:

Tentative Methods of Test for:

Sampling Paper and Paper Products (**D 585 – 40 T**),
Ash Content of Paper and Paper Products (**D 586 – 40 T**),
Casein in Paper (Qualitative) (**D 587 – 40 T**),
Alpha-, Beta-, and Gamma-Cellulose in Paper (**D 588 – 40 T**),
Paraffin Content of Waxed Paper (**D 590 – 40 T**), and
Starch in Paper (**D 591 – 40 T**)

These recommendations have been submitted to letter ballot of the committee which consists of 54 voting members; 37 members returned their ballots, with the results shown in Table I.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Paper Testing Methods (W. R. Willets, chairman) has held two meetings during the year and has continued its critical study of various methods of test, which have been approved by T.A.P.P.I., and is active in developing new methods of test.

The subcommittee has completed the following five new methods of test, which are appended to this report:[2]

Methods of Test for:

Pentosans in Paper,
Conditioning Paper and Paper Products for Testing,
Internal Tearing Resistance of Paper,
Quantitative Determination of Coating on Mineral-Coated Paper, and
Qualitative Examination of Mineral Filler and Mineral Coating of Paper.

The six tentative methods being recommended for adoption as standard, as mentioned earlier in this report, are under the jurisdiction of this subcommittee.

In addition, the subcommittee recommends that the Tentative Method of Test for Opacity of Paper and Paper Products (D 589 – 40 T) be retained as tentative. This method at present determines opacity by the contrast ratio. It is not suitable, however, for measuring printing opacity, and therefore its adoption as standard is not recommended at this time. Considerable work is now being carried on in an effort to revise this method as soon as possible to include a test procedure for printing opacity.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Test for Pentosans in Paper	29	0	8
Test for Conditioning Paper and Paper Products for Testing	34	0	3
Test for Quantitative Determination of Coating on Mineral-Coated Paper	28	0	9
Test for Internal Tearing Resistance of Paper	33	0	4
Test for Qualitative Examination of Mineral Fillers and Mineral Coatings of Paper	28	0	9
II. REVISIONS OF TENTATIVE STANDARDS			
Test for Basis Weight of Paper and Paper Products (D 646 – 41 T)	34	0	3
Test for Moisture in Paper (D 644 – 41 T)	34	0	3
III. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Test for Sampling of Paper and Paper Products (D 585 – 40 T)	33	0	4
Test for Ash Content of Paper and Paper Products (D 586 – 40 T)	30	0	7
Test for Casein in Paper (Qualitative) (D 587 – 40 T)	29	0	8
Test for Alpha-, Beta-, and Gamma-Cellulose in Paper (D 588 – 40 T)	29	0	8
Test for Paraffin Content of Waxed Paper (D 590 – 40 T)	30	0	7
Test for Starch in Paper (D 591 – 40 T)	30	0	7

At the present time, methods on the following subjects are being given consideration by Subcommittee I:

Surface Strength,
Heat Test for Relative Stability,
Water Vapor Permeability,
Tensile Strength,
Wire and Felt Side,
Bursting Strength,
Wet Tensile Strength,
Edge Tear,
Hydrogen Ion Concentration,
Curl,
Elongation,
Erasing Properties,
Stiffness,
Gloss,
Water Absorption,
Ply Adhesion,
Inflammability,
Turpentine Test for Grease Resistance of Paper, and
Alkali Staining Property of Paper.

In addition to the above, work has been completed on the following proposed methods, which have been referred to the Joint A.S.T.M.-T.A.P.P.I. Committee for action:

Kerosine Number for Roofing and Flooring Felts.
Air Resistance of Paper.

Subcommittee II on Significance of Test Methods (C. C. Heritage, chairman) held one meeting during the year at which further work was accomplished on the preparation of a monograph covering the significance of tests of paper and on definition of terms, nomenclature, and properties of paper. Several chapters have been prepared and submitted to the various members for criticism. It is planned to have another meeting of this subcommittee during the annual meeting of the Society in June. The work of this subcommittee has necessarily been delayed due to the conditions caused by the National Emergency.

Subcommittee IV on Fiberboard and Fiberboard Containers (R. L. Beach, chairman) has held no meetings during the year as the work of this subcommittee has had to be temporarily suspended during the National Emergency because the individual members directly concerned with the investigations which were in progress are devoting their entire time to the war effort. However, during the year, Subcommittee IV assisted Subcommittee I in revising the Tentative Method of Test for Moisture in Paper (D 644 – 41 T) so that it would also cover paperboard and fiberboard containers.

This being the year for election of officers, a Nominating Committee consisting of the chairmen of the Subcommittees of D-6, that is, Messrs. C. C. Heritage, R. L. Beach and W. R. Willets, was authorized to submit nominations for officers of the committee for the next two years. The following nominations were recommended and will be voted on at the meeting of Committee D-6 held during the annual meeting of the Society:

Chairman, L. S. Reid.
Vice-Chairman, M. A. Krimmel.
Secretary, G. H. Harnden

This report has been submitted to letter ballot of the committee, which consists of 54 voting members, 37 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

L. S. REID,
Chairman.

G. H. HARNDEN,
Secretary.

REPORT OF COMMITTEE D-7

ON

TIMBER

Committee D-7 on Timber held two meetings during the year in Chicago, Ill., one on June 26, 1941, and the other on March 17, 1942.

Revision of Standard, Immediate Adoption

The committee recommends for immediate adoption the revision of the Standard Method of Test for Distillation of Creosote (D 246 – 39) as appended hereto,[1] and accordingly asks for a favorable nine-tenths vote at the annual meeting in order that this recommendation may be referred to letter ballot of the Society. Subcommittee VI on Timber Preservatives (C. S. Reeve, chairman) prepared this revision which permits the use of an electric heater and means for controlling its operation, inasmuch as there has been a demand for such an alternate method, particularly for locations where gas is not available. The Preservatives Committee of the American Wood-Preservers' Association made a comprehensive comparison of the two methods of heating and the data in support of permitting the use of the specified electric heater were published in the 1940 *Proceedings* of the A.W.P.A. The recommended revision of Method D 246 brings it into accord with the present A.W.P.A. standard.

This recommendation has been submitted to letter ballot of the committee which consists of 24 members; 21 members returned their ballots of whom 19 have voted affirmatively, 0 negatively, and 2 members marked their ballots "not voting."

The various standards for timber and timber preservatives under the jurisdiction of this committee that have been published for a period of six years or more are being continued as standard without revision.

This report has been submitted to letter ballot of the committee which consists of 24 members; 18 members returned their ballots, of whom 17 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

Hermann von Schrenk,
Chairman.

J. A. Newlin,
Secretary.

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[1] This revised method was adopted as standard by the Society and appears in the 1942 Book of A.S.T.M. Standards, Part II, p. 679.

REPORT OF COMMITTEE D-8

ON

BITUMINOUS WATERPROOFING AND ROOFING MATERIALS

Committee D-8 on Bituminous Waterproofing and Roofing Materials has held two meetings during the past year: in Chicago, Ill., on June 24, 1941, and in Cleveland, Ohio, on March 5, 1942.

Subsequent to the 1941 annual meeting Committee D-8, jointly with Committee D-4 on Road and Paving Materials, presented to the Society through Committee E-10 on Standards the Tentative Volume Correction Table for Tar and Coal-Tar Pitch (D 633 - 41 T). This recommendation was accepted[1] by Committee E-10 on August 25, 1941, and the new tentative standard appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part II.

Committee D-8 also referred to Committee E-8 on Nomenclature and Definitions proposed tentative definitions for the terms Asphalt, Mineral Filler, Finely Powdered Mineral Filler, Rag Roofing Felt, Saturated, Dampproofing and Waterproofing, which were developed by its Subcommittee I on Nomenclature and Definitions (C. S. Reeve, chairman.)

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Prepared Roofings and Shingles (M. R. Beasley, chairman). —During the past year this subcommittee has been engaged in developing specifications for asphalt siding surfaced with coarse mineral granules and for insulated type asphalt siding surfaced with coarse mineral granules. It is expected that these will shortly be referred to letter ballot of Committee D-8 for recommendation to the Society. Subcommittee II has also cooperated with Committee D-6 on Paper and Paper Products in developing a satisfactory procedure for determining kerosine number for roofing and flooring felt.

EMERGENCY ALTERNATE PROVISIONS

Committee D-8 has recommended to Committee E-10 on Standards that under the established procedure for emergency requirements a sticker be attached to the Tentative Specifications for Asphalt Roofing Surfaced with Coarse Mineral Granules (D 249 - 41 T) and Tentative Specifications for Asphalt Roofing Surfaced with Powdered Talc or Mica (D 224 - 41 T) providing that the requirement for protective coating on nails furnished with the roofing may be suspended. These Emergency Alternate Provisions were accepted by Committee E-10 on May 11, 1942, and have been published as stickers for attachment to the specifications to which they apply.

I. NEW TENTATIVE STANDARDS

Committee D-8 recommends that the following two new tentative specifications developed by its Subcommittee IV on Membrane Waterproofing and Built-

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[1] In submitting this recommendation to Committee E-10 on Standards, Committee D-8 reported the following results of the letter ballot vote of a total of 32 ballots returned from a committee membership of 59: Affirmative 30, negative 0, and 2 ballots marked "not voting."

Up Roofing (E. H. Berger, chairman) be accepted for publication as tentative:

Tentative Specifications for:

Coal-Tar Pitch for Steep Built-Up Roofs[2] (published as an appendix to the 1941 annual report of Committee D-8), and

Asphalt Saturated and Asphalt Coated Asbestos Felts for Use in Constructing Built-Up Roofs.[3]

II. REVISION OF TENTATIVE STANDARD

Tentative Specifications for Asphalt for Dampproofing and Waterproofing (**D 449 - 37 T**).[4]—Committee D-8 feels that these specifications should be expanded to include a third type of asphalt suitable for exposure temperatures above 125 F. (52 C.). The committee therefore recommends that this tentative standard be revised as follows and continued as tentative:

Section 4.—Change the introductory sentence to indicate that three types of asphalt are covered by these specifications rather than two and add the following paragraph covering Type C:

> *Type C.*—An asphalt less susceptible to temperature than Type B, with good adhesive properties for use above ground level where exposed on vertical surface in direct sunlight or at temperatures above 125 F. (52 C.).

Section 5.—In Table I, add the following requirements for Type C:

	Type C	
	Min.	Max.
Softening point (ring-and-ball method)	170 F.	200 F.
Flash point (Cleveland open cup)	400 F. (205 C)	...
Penetration:		
0 C. (32 F.), 200 g., 60 sec.	10	...
25 C. (77 F.), 100 g., 5 sec.	20	40
46 C. (115 F.). 50 g., 5 sec.	...	100
Ductility at 25 C. (77 F.) (5 cm. per min.), cm.	2.5	...
Loss on heating at 163 C. (325 F.), 50 g., 5 hr., per cent.	...	1
Penetration of residue, percent of original	60	...
Total bitumen soluble in carbon disulfide, per cent	99	...
Proportion of bitumen soluble in carbon tetrachloride, per cent	99	...
Ash, per cent(unfilled asphalt).	...	1

III. REVISION OF STANDARD, IMMEDIATE ADOPTION

Standard Specifications for Coal-Tar Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (**D 227 - 41**).[5]—In order to bring these specifications in line with a proposed Federal Specification, Committee D-8 recommends that the following revision be incorporated in this standard without publication as a tentative revision. The committee accordingly requests the necessary nine-tenths affirmative vote at the annual meeting in order that this recommendation may be referred to letter ballot of the Society:

Section 3.—Change the last sentence to read as follows by the omission of the words in brackets:

> The surface of the felt shall be uniformly smooth. Upon splitting or tearing on the bias, the felt shall appear reasonably free from lumps of underbeaten stock (that is, stock which has not been beaten or shredded into fiber in the process of manufacture) and particles of foreign substances (that is, fragments of stone, metal, leather, rubber, [straw, wood,] etc.).

Section 7.—Change the requirement for pliability to read as follows by the addition of the italicized figure, and the omission of the figure in brackets.

Pliability at 77 F. (25 C.)	at least *10* [8] out of 10 strips shall not crack when bent 90 deg. at a uniform speed over a rounded corner of ½-in. radius.

IV. ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the following tentative standards be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated:

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[2] These specifications were accepted as tentative by the Society and appear in the 1942 Book of A.S T.M. Standards, Part II, p. 1293.

[3] These specifications were accepted as tentative by the Society and appear in the 1942 Book of A.S.T.M. Standards, Part II, p. 1301.

[4] 1939 Book of A.S.T.M. Standards, Part II, p. 1059.

[5] 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 144.

Tentative Specifications for Asphalt-Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (**D 226 - 41 T**):[6]

Section 4.—Make the same change in this section as is recommended above in Section 3 of Specifications D 227 – 41.

Section 6.—In the table change requirement for pliability to read as follows by the addition of the italicized figure and the omission of the figure in brackets:

Pliability at 77 F. (25 C.)...At least *10* [8] strips out of 10 shall not crack when bent 90 deg. at a uniform speed over a rounded corner

In the table change item on breaking strength to read as follows by the addition of the italicized word and figures:

Average breaking strength *at 77 F. (25 C.)*

Tentative Specifications for Woven Cotton Fabrics Saturated with Bituminous Substances for Use in Waterproofing (**D 173 - 40 T**):[7]

Section 6.—Change to read as follows by the addition of the italicized figure and the omission of the figure in brackets:

6. The selvage shall not measure over *1/4* $\frac{3}{16}$] in.

Tentative Methods of Testing Films Deposited from Bituminous Emulsions (**D 466 - 38 T**):[8]

Section 2 (a).—Change to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

(*a*) Brass Mask.—A brass mask approximately $\frac{7}{64}$ in. in thickness with a rectangular opening *4 by 4 in.* [of at least 12 sq. in.].

Section 5 (b).—Change third and last sentences to read as follows by the addition of the italicized words and the omission of the word in brackets:

After 24 hr. the character of the bituminous film shall be noted by cutting *and attempting to ltfi a portion of the submerged area* with a knife or teasing needle.

Re-emulsification is indicated if the water becomes darkened by rubbing the *submerged* [wet] surface of the *uncut* film lightly with a rubber policeman.

V. Revision and Reversion to Tentative of Standard

Standard Specifications for Asphalt-Saturated Asbestos Felt for Use in Constructing Built-Up Roofs (**D 250 - 27**).—A tentative revision of these specifications was issued in 1939. During the past year these specifications have been further expanded and completely revised, and Committee D-8 now recommends that the present standard specifications be reverted to tentative as revised. The revised specifications are appended hereto.[9]

VI. Withdrawal of Tentative Standard

Tentative Specifications for Asphalt Roofing Surfaced with Fine Mineral Granules (**D 248 - 41 T**). — Because it has been found that roofing meeting these specifications is manufactured as a specialty by only one or two producers, Committee D-8 recommends that they be withdrawn.

VII. Editorial Changes in Standards

Standard Specifications for Creosote for Priming Coat with Coal-Tar Pitch in Dampproofing and Waterproofing (**D 43 - 41**):[10]

Section 2.—In the tabulation add a footnote reference letter *a* following the words "Consistency at 5 C. (41 F.)" and add a new footnote *a* to read as follows:

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[6] 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 354.

[7] 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 305.

[8] 1939 Book A.S.T.M. Standards, Part II, p. 1068.

[9] This recommendation was accepted by the Society and the revised specifications appear in the 1942 Book of A.S T.M. Standards, Part II, p 1299.

[10] 1941 Supplement to Book of A.S.T.M. Standards, Part II, pp. 134, 366, 369, 360, 357.

[a] The creosote shall be rated as crystal free, if no crystals are formed when 100 ml. of the sample are maintained at a temperature of 5 C. (41 F.) for 3 hr. in a 125-ml. Erlenmeyer flask with occasional stirring.

Tentative Specifications for Asphalt Roofing Surfaced with Powdered Talc or Mica (**D 224 – 41 T**):[10]

Section 3.—Change the second sentence from its present form to read as follows:

> The surface of the felt shall be uniformly smooth. Upon splitting or tearing on the bias, the felt shall appear reasonably free from lumps of underbeaten stock (that is, stock which has not been beaten or shredded into fiber in the process of manufacture) and particles of foreign substances (that is, fragments of stone, metal, leather, rubber, etc.).

Section 4.—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

> 4. The *bitumen of the* saturant and coatings shall be composed [principally] of asphaltic materials.

Tentative Specifications for Asphalt Shingles Surfaced with Coarse Mineral Granules (**D 225 – 41 T**):[10]

Sections 3 and 4.—Make the same change in these sections as is recommended above in Sections 3 and 4 of Specifications D 224 – 41 T.

Tentative Specifications for Asphalt Roofing Surfaced with Coarse Mineral Granules (**D 249 – 41 T**):[10]

Sections 3 and 4.—Make the same change in these sections as is recommended above in Sections 3 and 4 of Specifications D 224 – 41 T.

Tentative Specifications for Wide Selvage Asphalt Roofing Surfaced with Coarse Mineral Granules (**D 371 – 41 T**):[10]

Sections 3 and 4.—Make the same change in these sections as is recommended above in Sections 3 and 4 of Specifications D 224 – 41 T.

Tentative Methods of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing (**D 146 - 38 T**).[11]—Committee D-8 recommends that the following revision be incorporated in these methods and accepted as an editorial change. This change makes the methods consistent with certain existing A.S.T.M. specifications which refer to this tentative standard.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Spec. for Coal-Tar Pitch for Steep Built-Up Roofs........	26	0	10
Spec. for Asphalt Saturated and Asphalt Coated Asbestos Felts for Use in Constructing Built-Up Roofs..........	30	0	6
II. REVISION OF TENTATIVE STANDARD			
Spec. for Asphalt for Dampproofing and Waterproofing (D 449 – 37 T)...............	33	1	2
III. REVISION OF STANDARD, IMMEDIATE ADOPTION			
Spec. for Coal-Tar Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (D 227 – 41).................	26	0	10
IV. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Spec. for Asphalt-Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (D 226 – 41 T)..............	30	0	6
Spec. for Woven Cotton Fabrics Saturated with Bituminous Substances for Use in Waterproofing (D 173 – 40 T)..........................	30	0	6
Methods of Testing Films Deposited from Bituminous Emulsions (D 466 – 38 T)....	30	1	5
V. REVISION AND REVERSION TO TENTATIVE OF STANDARD			
Spec. for Asphalt-Saturated Asbestos Felt for Use in Constructing Built-Up Roofs (D 250 – 27)..........	30	0	6
VI. WITHDRAWAL OF TENTATATIVE STANDARD			
Spec. for Asphalt Roofing Surfaced with Fine Mineral Granules (D 248 – 41 T).....	30	0	6

Section 12 (a).—In the third sentence change the temperature at which the specimens are tested from "70 F. (21.1 C.)" to read "77 F. (25 C.)."

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[11] 1939 Book of A.S.T.M. Standards, Part II, p. 1107.

Tentative Methods of Testing Asphalt Roll Roofing, Cap Sheets, and Shingles (**D 228 - 37 T**).[12]—The committee recommends that the following change be incorporated in these methods and accepted as a correction of a typographical error.

Section 25 (*a*).—In the table on reporting of results change the first item to read as follows by the addition of the italicized figures and the omission of those in brackets:

Passing Sieve	Retained on Sieve	Weight of Mineral Matter per 108 sq. ft.
No. 6 (3360-micron)	No. *14* [100] (*1410*-[149] micron)	....

VIII. STANDARD AND TENTATIVE STANDARD CONTINUED WITHOUT REVISION

Committee D-8 recommends that the following method of test and recommended practice be continued without change:

Standard Method of Test for Steam Distillation of Bituminous Protective Coatings (D 255 - 28), and

Tentative Recommended Practice for Accelerated Weathering Test for Bituminous Materials (D 529 - 39 T).

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 58 voting members; 36 members returned their ballots with the results shown in Table I.

At a meeting on June 23, 1942, the following officers were elected for the ensuing term of two years:

Chairman, J. M. Weiss.
Vice-Chairman, E. H. Berger.
Secretary, Prévost Hubbard.

This report has been submitted to letter ballot of the committee which consists of 58 voting members; 36 members returned their ballots, of whom 33 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. M. WEISS,
Chairman.

PRÉVOST HUBBARD,
Secretary.

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[12] 1939 Book of A.S.T.M. Standards, Part II, p. 1115.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee D-8 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative Specifications for:
Asphalt Siding Surfaced with Coarse Mineral Granules (D 699 - 42 T),

Revision of Tentative Specifications for:
Asphalt Roofing Surfaced with Coarse Mineral Granules (D 249 - 41 T), and

Revision of Tentative Methods of:
Testing Asphalt Roll Roofing, Cap Sheets, and Shingles (D 228 - 37 T).

These recommendations were accepted by Committee E-10 on November 9, 1942, and the new and revised tentative standards appear in the 1942 Book of A.S.T.M. Standards, Part II, pp. 1317, 1307, and 1328.

REPORT OF COMMITTEE D-9

ON

ELECTRICAL INSULATING MATERIALS

Committee D-9 on Electrical Insulating Materials held two meetings during the year and will hold a third during the annual meeting of the Society in June. One of the meetings was held in New York, N. Y., on September 29 and 30, 1941, and the other in Philadelphia, Pa., on February 17 and 18, 1942. These meetings were held in conjunction with the meetings of Committee D-20 on Plastics because of the close cooperation between the two committees and the large number of members who serve on both committees.

At these meetings recommendations were made which resulted in additions to or changes in some of the standards for which Committee D-9 is responsible. Other projects which are still under way are mentioned later in this report.

Subsequent to the 1941 annual meeting, Committee D-9 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative Specifications for:

Molds for Test Specimens of Molding Materials Used for Electrical Insulation (D 647 - 41 T),

New Tentative Methods of Test for:

Product Uniformity of Phenolic Laminated Sheets (D 634 - 41 T),

Revisions of Tentative Methods of:

Sampling and Testing Untreated Paper Used in Electrical Insulation (D 202 - 40 T),

Test for Thickness of Solid Electrical Insulation (D 374 - 36 T),

Tentative Revisions of Standard Methods of:

Testing Molded Materials Used for Electrical Insulation (D 48 - 39), in the form of the following five new tentative methods:

Testing Molded Materials Used for Electrical Insulation (D 48 - 41 T),

Test for Distortion Under Heat of Molded Electrical Insulating Materials (D 648 - 41 T),

Test for Compressive Strength of Electrical Insulating Materials (D 649 - 41 T),

Test for Flexural Strength of Electrical Insulating Materials (D 650 - 41 T),

Test for Tensile Strength of Electrical Insulating Materials (D 651 - 41 T),

Testing Shellac Used for Electrical Insulation (D 411 - 40), and

Test for Impact Resistance of Electrical Insulating Materials (D 256 - 38), in the form of new Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (D 256 - 41 T).

These recommendations were accepted[1] by Committee E-10 on August 25, 1941.

On October 22, 1941, Committee E-10 accepted[1] for publication new Tentative Methods of Measuring Mica Stampings Used in Electronic Devices and Incandescent Lamps, which bear the A.S.T.M. serial designation D 652 - 41 T.

A revision of the Tentative Method of Test for Power Factor and Dielectric Constant of Natural Mica was approved[1] by Committee E-10 on January 26, 1942. This method as revised now bears the designation D 351 - 42 T.

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee D-9 reported the following results of the letter ballot vote of a total of 55 ballots returned from a committee membership of 96: D 647, affirmative 26, negative 2, ballots marked "not voting" 27; D 634, affirmative 22, negative 0, ballots marked "not voting" 33; D 202, affirmative 28, negative 0, ballots marked "not voting" 27; D 374, affirmative 36, negative 0, ballots marked "not voting" 19; D 48, D 648, D 649, D 650, and 651, affirmative 26, negative 2, ballots marked "not voting" 27; D 411, affirmative 23, negative 0, ballots marked "not voting" 32; D 256, affirmative 30, negative 0, ballots marked "not voting" 25; D 652, 63 ballots returned: affirmative 26, negative 0, ballots marked "not voting" 37; D 351, 62 ballots returned: affirmative 30, negative 0, ballots marked "not voting" 32.

RECOMMENDATIONS AFFECTING STANDARDS

As a result of the year's work, the committee is submitting five new tentative standards, revisions of four tentative standards and two standards, tentative revisions of five standards, and recommending the adoption as standard of three tentative standards, the adoption of revisions in five standards, the withdrawal of two standards, and editorial changes in an existing standard. The standards affected are listed in Table I. The recommendations being presented are included in Appendix I, each proposed revision being accompanied by a brief reason for the revision.

These recommendations have been submitted to letter ballot of the committee which consists of 96 voting members, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Test for Sludge Formation in Mineral Transformer Oil[a]	21	5	30
Testing Steatite Used as Electrical Insulation	23	0	33
Testing Vulcanized Fiber Used for Electrical Insulation (to replace Tentative Methods D 619 - 41 T)	24	0	32
Measuring Dimensions of Rigid Tubes Used in Electrical Insulation	27	0	29
Test for Power Factor and Dielectric Constant Parallel with Laminations of Laminated Sheet and Plate Insulating Materials	31	0	25
II. REVISIONS OF TENTATIVE STANDARDS			
Spec. for Molds for Test Specimens of Molding Materials Used for Electrical Insulation (D 647 - 41 T)	24	0	32
Test for Compressive Strength of Electrical Insulating Materials (D 649 - 41 T)		0	25
Test for Flexural Strength of Electrical Insulating Materials (D 650 - 41 T)	30	0	25
Test for Tensile Strength of Electrical Insulating Materials (D 651 - 41 T)	31	0	25
III. REVISION OF STANDARD, IMMEDIATE ADOPTION	31		
Test for Arc Resistance of Solid Electrical Insulating Materials (D 495 - 41)	30	0	26
Testing Electrical Insulating Oils (D 117 - 40; ASA C59.2-1941)	24	0	31
IV. TENTATIVE REVISIONS OF STANDARDS			
Testing Electrical Porcelain (D 116 - 39)	24	0	32
Testing Sheet and Plate Materials Used in Electrical Insulation (D 229 - 39)	27	0	29
Testing Laminated Tubes Used in Electrical Insulation (D 348 - 39)	26	0	30
Testing Laminated Round Rods Used in Electrical Insulation (D 349 - 39)	25	0	31
Testing Shellac Used for Electrical Insulation (D 411 - 40)	23	0	33
V. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Test for Thickness of Solid Electrical Insulation (D 374 - 41 T)	30	0	26
Testing Pin-Type Lime Glass Insulators (D 468 - 39 T),[b] as revised	22	3	31
Test for Water Absorption of Plastics (D 570 - 40 T)	27	0	22
VI. ADOPTION AS STANDARD OF TENTATIVE REVISIONS OF STANDARD			
Testing Electrical Porcelain (D 116 - 39)	23	2	31
Testing Sheet and Plate Materials Used in Electrical Insulation (D 229 - 39)	28	0	28
Testing Laminated Tubes Used in Electrical Insulation (D 348 - 39)	26	0	30
Testing Laminated Round Rods Used in Electrical Insulation (D 349 - 39)	25	0	31
Testing Shellac Used for Electrical Insulation (D 411 - 40)	24	0	32
VII. WITHDRAWAL OF STANDARDS			
Testing Molded Materials Used for Electrical Insulation (D 48 - 39; C59.1-1940)	2	0	31
Test for Impact Resistance of Electrical Insulating Materials (D 256 - 38; C59.11-1941)	2	0	29
VIII. EDITORIAL CHANGE IN STANDARD			
Testing Sheet and Plate Materials Used in Electrical Insulation (D 229 - 39)	29	0	27

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[a] The classified vote on the proposed Tentative Methods of Test for Sludge Formation in Mineral Transformer Oil was as follows: Affirmative: 6 producers, 12 consumers, 3 general interests; negative: 5 producers, 0 consumers, 0 general interests.

[b] The classified vote on the adoption as standard of the Tentative Methods of Testing Pin-Type Lime-Glass Insulators, (D 468 - 39 T), as revised, was as follows: Affirmative: 3 producers, 13 consumers, 6 general interests; negative: 3 producers, 0 consumers, 0 general interests.

ACTIVITIES OF SUBCOMMITTEES

Some of the projects now in progress are briefly mentioned in the following summary of subcommittee activities:

Subcommittee I on Insulating Varnishes, Paints and Lacquers (C. F. Hanson, chairman). — Work is now in progress looking toward the establishment of purchase specifications for both orange shellac and shellac varnish used for electrical insulation.

Another round robin test of acid and alkali resistance has been completed. Test results from various laboratories were widely inconsistent. The consensus is that test specimens would have to be prepared with most meticulous care in an atmosphere entirely free of dust. These conditions are difficult to fulfill for commercial purposes. A survey is under way to find ways of approaching the problem from another angle.

A section has been organized to prepare a description of the significance of the various tests described in the Standard Methods of Testing Varnishes Used for Electrical Insulation (D 115 - 41).

A method for determining the internal drying of a saturant varnish is still under consideration. An effort is being made to develop a test under a condition which simulates conditions existing in practice. When this procedure is attempted several problems arise which are not easy to solve.

Subcommittee II on Molded Insulating Materials (W. A. Evans, chairman).—Proposed specifications for phenolic molding materials which provide four classifications of materials have been prepared It is felt, however, that the classification should be extended to include seven classifications as in the Navy specification. After this draft has been revised it will be distributed for comment.

Specified values for the electrical tests on phenolic materials are being prepared for comment. Where values are not available, specimens will be submitted to round robin tests from which suggested values may be obtained.

Subcommittee III on Plates, Tubes and Rods (Dean Harvey, chairman).—In order to simplify and facilitate the work of Subcommittee III, the organization of the sections has been studied and consolidations are being made. A new Section 1 on Mechanical Properties, under the chairmanship of W. A. Zinzow, has taken over the work of four former sections; a new Section 2 on Electrical Properties, with S. W. Place, as chairman, has replaced two former sections. Other changes are under consideration. Mr. G. H. Mains has been appointed vice-chairman of the subcommittee.

The Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (D 256-41 T), prepared jointly by Committee D-9 and Committee D-20 on Plastics have been approved by the subcommittee.

A study is being made of the significance of the various tests which have been developed, and statements are being prepared.

The preparation of specifications for the various materials coming within the scope of the subcommittee has been under consideration, and a study is being made to determine those which are most needed by the Government and other organizations in the war program.

Considerable progress has been made in the study of the flexural strength test to determine the effect of various factors upon the accuracy of this test. A report on power factor measurements parallel with laminations of phenolic laminated sheet has been prepared by Section O and appears in Appendix II.

Subcommittee IV on Liquid Insulation (E. A. Snyder, chairman).—Committee D-2 on Petroleum Products and Lubricants has recommended that the Methods of Test for Neutralization Number of Petroleum Products and Lubricants (D 188 - 41 T) be withdrawn and replaced by the Proposed Tentative Methods of Test for Neutralization Number of Petroleum Products by Electrometric Titration and by Color-Indicator Titration. Tests will be conducted to determine whether these new proposed methods are satisfactory for insulating oils and, if so, they will be adopted in place of Methods D 188 - 41 T.

Although the Methods of Test for Sludge Formation in Mineral Transformer Oil[2] are being submitted as a tentative standard, it should be noted that the work to date has resulted merely in developing test procedures which give checkable test results but that the coordination of these test results with actual operating conditions has, as yet, not been developed. A historical survey of the development of the sludge test methods from 1926 to 1942 has been prepared by A. E. Flowers and is presented in an appendix[3] to this report. This survey shows the enormous amount of work performed by the subcommittee and the difficult task it had in eliminating the discrepancies in test results between cooperating laboratories.

On recommendation of the subcommittee, Committee D-9 has accepted the name "Askarel" adopted by the National Electrical Manufacturers Association to designate synthetic noninflammable insulating liquids. Proposed methods of test have been prepared covering all characteristics of Askarels except free chlorides, refractive index, dielectric strength, and analysis of arc gases. Procedures for these four tests are now being developed.

The proposed method of determining dielectric strength of insulating oils issued as a tentative revision[4] of Standard Method D 117 - 40 is being continued as tentative, pending a revision of the procedure or method of mixing the sample prior to making the dielectric strength test.

The significance of the tests for color, neutralization number, dielectric strength, and mineral acids has been approved and submitted to Subcommittee XI on Significance of Tests.

The section working on the development of a test to determine quantitatively small amounts of moisture in insulating oil is conducting round robin tests on a number of proposed test methods and is making satisfactory progress.

The section developing test methods to determine the gas content of oil is making progress on improving the apparatus in respect to methods of heating the sample, measuring the vacuum and condensing vapors. Progress is also being made in designing suitable containers for satisfactorily maintaining the original gas content of the oil.

Subcommittee V on Ceramic Products (Porcelain, Glass, Etc.) (L. J. Cavanaugh, chairman).—The results of a recent series of round robin tests on compressive strength of steatite have been received. Although all data have not been tabulated, the results indicate satisfactory agreement between laboratories when using soft steel cushions in place of blotting paper cushions.

A new section is being appointed to formulate product specifications for steatite.

The American Institute of Electrical Engineers is being asked to make provisions for lime-glass insulators in their proposed standard on insulator tests.

Subcommittee VI on Solid Filling and Treating Compounds (R. H. Titley, chairman).—The section working on the problem of a method of determining the solubility of filling and treating compounds in oil, reports progress. Up to the present no suitable accelerated test has been developed but it is hoped that one can be obtained in the near future.

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[2] See p. 371.

[3] See p. 896.

[4] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 567; also 1941 Supplement, Part III, p. 632.

As a result of discussion in the subcommittee, it does not seem advisable to recommend the adoption as standard at this time of the Tentative Methods of Testing Solid Filling and Treating Compounds Used for Electrical Insulation (D 176 - 40 T).

Subcommittee VII on Electrical Tests (J. A. Scott, chairman).—Work has continued on thermal conductivity tests. A method based on the customary hot plate arrangement was drawn up, but action was withheld pending study of two more rapid methods—one the Dannatt-Metropolitan Vickers method and the other the Cenco-Fitch method.

The Section on Insulation Resistance has become interested in current time curves. While recognizing the difficulty at present of using these in routine tests, their study should be informative.

Recommendations on cells for power factor tests on cable oils and the handling of samples were given informally to Subcommittee IV, and assistance will be continued to prepare these for inclusion in the oil test methods.

Subcommittee VIII on Insulating Papers and Fabrics (R. W. Chadbourn, chairman).—Work is progressing jointly between Committee D-9 and Committee D-6 on Paper and Paper Products in the determination of a method for obtaining the pH value of paper samples. The reason for the previous discrepancies has apparently been determined and a proposed method of test is to be circulated for approval for conducting the round robin tests.

Work has been progressing on reviewing methods of mechanical tests for papers proposed by Committee D-6. However, it has been tentatively agreed that it is undesirable to attempt to accept individual methods as they are completed by Committee D-6 but to wait until all of its methods are completed and review them as a whole.

Consideration is being given to a dielectric strength test of varnished cloth tape while the tape is elongated. Proposed revisions based on round robin test data are to be submitted by manufacturers and consumers.

A round robin test has been completed on the rate of burning of varnished tubing using both the A.S.T.M. Methods of Testing Flexible Varnished Tubing Used for Electrical Insulation (D 350 - 40 T) and Underwriters' Tentative Specification Subject No. 746. The results showed that the A.S.T.M. method was more consistent than the Underwriters' method and should be retained. It was also agreed to retain the dielectric strength requirements in Methods D 350 - 40 T and the A.S.T.M. Tentative Specifications for Flexible Varnished Tubing Used in Electrical Insulation (D 372 - 40 T) in preference to the Underwriters' requirements.

In connection with the testing of treated glass tapes and fabrics, consideration is being given to the use of the methods for varnished cloth tape and it appears that this will be possible in most cases.

Subcommittee IX on Mica Products (M. P. Davis, chairman).—A list of diameters of punched mica stampings now being commercially used is being compiled as a basis for establishing a dimensional grading chart of small mica films. After several years of trial by various photographers, one of them has succeeded in satisfactorily duplicating the mica samples with color transparencies of the various classifications of mica.

Subcommittee X on Conditioning (Robert Burns, chairman).—A review has been made of the principal items in U. S. Government specifications which would be considered nonstandard by A.S.T.M. Although the conditioning procedures in Government specifications present the same problem as A.S.T.M. methods so far as the multitude of special procedures are concerned, there are certain general features in the former which might be brought into conformance with A.S.T.M. methods with little or no sacrifice in adequacy. It was agreed that these would be discussed with the proper authorities.

The subcommittee has discussed the integration of the various conditioning standards of Committees D-9 and D-20 with those of other standing committees through the efforts of Technical Committee X on Conditioning and Weathering, of Committee E-1 on Methods of Testing. The standard definitions and standard laboratory atmosphere proposed by this subcommittee are now under review by the E-1 technical committee.[5] Also under review is the National Bureau of Standards sun lamp —dew accelerated weathering test submitted through this committee. This is not a standard, but is being reviewed at the request of this subcommittee as a supplement to the so-called weatherometer type test.

Statements are being prepared on the significance of the various conditioning tests.

Subcommittee XI on Significance of Tests (W. A. Zinzow, chairman).—The several subcommittee chairmen of the other subcommittees of Committee D-9 have been asked to appoint sections to prepare statements for insertion in the various standards and tentative standards under their jurisdiction regarding the significance of the test or test procedures covered by those methods. Preparation of some of these statements is actively under way in some of the subcommittees and when they have been completed and approved by the respective subcommittees they will be reviewed by this subcommittee before submittal to Committee D-9 for consideration.

The following officers of the committee were unanimously elected for the ensuing term of two years:

Chairman, E. J. Rutan.
Vice-Chairman, W. A. Evans.
Secretary, W. A. Zinzow.

This report has been submitted to letter ballot of the committee which consists of 96 voting members; 55 members returned their ballots, of whom 45 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

T. SMITH TAYLOR,
Chairman.

E. J. RUTAN,
Secretary.

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[5] See p. 462.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee D-9 presented to the Society through Committee E-10 on Standards the following recommendations:

Revisions of Tentative Methods of:

Testing Molded Materials Used for Electrical Insulation (D 48 - 41 T),

Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (D 150 - 41 T), and

Testing Solid Filling and Treating Compounds Used for Electrical Insulation (D 176 - 40 T).

These recommendations were accepted by Committee E-10 on August 24, 1942, and the revised tentative methods appear in the 1942 Book of A.S.T.M. Standards, Part III, pp. 1055, 1148 1126.

APPENDIX I

RECOMMENDATIONS AFFECTING STANDARDS FOR ELECTRICAL INSULATING MATERIALS

In this Appendix are given recommendations affecting certain standards and tentative standards covering electrical insulating materials which are referred to earlier in this report.[1] The standards and tentative standards appear in their present form in either the 1939 Book of A.S.T.M. Standards, Part III, or the 1940 and 1941 Supplement, Part III as indicated by the final number in the A.S.T.M. Designation.

NEW TENTATIVE STANDARDS

Tentative Methods of Test for Sludge Formation in Mineral Transformer Oil:[2]

In the 1941 Report of Committee D-9 a sludge accumulation test and a high-pressure oxidation test for sludge formation in mineral transformer oil were published as information. It is now recommended that these methods be accepted for publication as tentative.

Tentative Methods for Testing Steatite Used as Electrical Insulation:[3]

These methods outline procedures for making various tests for an investigation or examination of steatite used as electrical insulation.

Tentative Methods of Testing Vulcanized Fibre Used for Electrical Insulation:[4]

These methods include several new procedures for the testing of vulcanized fibre sheets, tubes, and rods, and also procedures formerly appearing in the Standard Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (D 229–39), as well as the test for volatile matter, slightly modified, appearing in the Tentative Method of Test for Volatile Matter Content of Vulcanized Fibre (D 619–41 T).

Tentative Methods for Measuring Dimensions of Rigid Tubes Used in Electrical Insulation:[5]

These methods were prepared to insure more consistent procedure and results in measuring the dimensions of round tubes.

Tentative Method of Test for Power Factor and Dielectric Constant Parallel with Laminations of Laminated Sheet and Plate Insulating Material:[6]

This method was prepared subsequent to a series of round-robin tests in which various procedures were tried. The development of this method of test is described in a report on Data on Power Factor Measurements Parallel with Laminations of Phenolic Laminated Sheet, which appears in Appendix II.

REVISIONS OF TENTATIVE STANDARDS

Tentative Specifications for Molds for Test Specimens of Molding Materials Used for Electrical Insulation (D647-41T):

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[1] See p. 366.

[2] These methods were accepted as tentative by the Society and appear in the 1942 Book of A.S.T.M. Standards, Part III, p. 1114.

[3] These methods were accepted as tentative by the Society and appear in the 1942 Book of A.S.T.M. Standards, Part III, p. 1124.

[4] These methods were accepted as tentative by the Society and appear in the 1942 Book of A.S.T.M. Standards, Part III, p. 1090.

[5] These methods were accepted as tentative by the Society and appear in the 1942 Book of A.S.T.M. Standards, Part III, p. 1096.

[6] This method was accepted as tentative by the Society and appears in the 1942 Book of A.S.T.M. Standards, Part III, p. 1179.

This revision consists of a minor change in the scope clause of the specifications and the addition of requirements for a mold for tension test specimens.

Section 1.—In the last sentence and in the note following this section change "manufacturer" to read "seller of the material."

New Section.—Add a new Section 4 to read as follows:

> 4. *Molds for Tension Test Specimen.*—Molds for the tension test specimen may be of the design and dimensions shown in Fig. 4.[6a]

Tentative Method of Test for Compressive Strength of Electrical Insulating Materials (D 649 – 41 T):

This revision consists of a minor change in the scope clause so that it will not conflict with the procedures described in the Standard Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (D 229 – 39).

Section 1.—Change from its present form to read as follows:

> 1. This method of test covers a general procedure for determining the compressive strength of various solid electrical insulating materials. However, modifications are found desirable for different types of such materials and existing methods of the American Society for Testing Materials covering the requirements for test of a given material should be consulted for special details that may apply.

Tentative Method of Test for Flexural Strength of Electrical Insulating Materials (D 650 – 41 T):

This revision consists of a minor change in the scope clause so that it will not conflict with the procedures described in the Standard Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (D 229 – 39).

Section 1.—Change from its present form to read as follows:

> 1. This method of test covers a general procedure for determining the flexural strength of various solid electrical insulating materials. However, modifications are found desirable for different types of such materials and existing methods of the American Society for Testing Materials covering the requirements for test of a given material should be consulted for special details that may apply.

Tentative Method of Test for Tensile Strength of Electrical Insulating Materials (D 651 – 41 T):

The revision of this method consists of modifications in the title and wording of the method so that it will apply only to molded electrical insulating materials. The method in its revised form is appended hereto.[7]

REVISION OF STANDARD, IMMEDIATE ADOPTION

Standard Method of Test for Arc Resistance of Solid Electrical Insulating Materials (D 495 – 41):

The use of the present method has shown the need for certain minor changes principally in the scope of the test. The committee recommends for immediate adoption the following revisions and accordingly asks for a favorable nine-tenths vote at the annual meeting in order that these modifications may be referred to letter ballot of the Society:

Section 1.—Change the third sentence to read as follows by the addition of the italicized words and figure and omission of those in brackets:

> *For at least the first three steps of the test, when the testing current is only 10 milliamperes,* differences [in arc resistance] must be of the order of 2 to 1 (in seconds) in order to be significant.

In the first item in the table change "60 sec. and under" to read "30 sec. and under" for hot-molded or laminated materials (general).

[6a] This figure has been added to Tentative Specifications D 647 which appear in their latest revised form in the 1942 Book of A.S.T.M. Standards, Part III, p. 1049. Blueprints of detailed drawings for the construction of this mold will be obtainable at a nominal cost from the American Society for Testing Materials, 260 S. Broad St., Philadelphia, Pa.

[7] This revised method was accepted as tentative by the Society and appears in its latest revised form in the 1942 Book of A.S.T.M. Standards, Part III, p. 1077.

Section 4.—Change to read as follows by the addition of the italicized words:

4. The test specimens may be of any size or shape, provided a flat, or nearly flat, surface of about 0.16 sq. in. (1 sq. cm.) in area *and at least ⅛ in. in thickness, either in one piece or in a pile-up of thin sheets,* is available for each test.

Section 7.—Add new items (*1*) and (*4*) to be reported to read as follows, relettering the present items (*1*) and (*2*) as (*2*) and (*3*), respectively:

(*1*) A suitable description of the material tested and the approximate thickness at the test location.

(*4*) A general statement describing the behavior of the material during the test and the manner in which it fails (carbonizes, volatilizes, fuses, melts, or similar reaction).

Standard Methods of Testing Electrical Insulating Oils (D 117 - 40):

Committee D-2 on Petroleum Products and Lubricants has prepared a method of determining kinematic viscosity and a method for conversion of kinematic viscosity to Saybolt universal viscosity. It is recommended that reference be made to these two methods in Section 11 of Methods D 117 as indicated below.

Section 11.—Change to read as follows by the addition of the italicized words and figure and the omission of the words in brackets:

11. Viscosity shall be determined in accordance with *either* the Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (A.S.T.M. Designation: D 88) *or the Tentative Method of Test for Kinematic Viscosity (A.S.T.M. Designation: D 445)* of the American Society for Testing Materials, and unless otherwise [directed] *specified,* [shall be taken] at a temperature of 37.8 C. (100 F.). *The conversion of viscosity values may be made in accordance with the Standard Method for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity (A.S.T.M. Designation: D 446) of the American Society for Testing Materials.*

New Section.—Add a new Section 15 to provide for the determination of saponification number to read as follows:

15. *Saponification Number: Procedure.*—Saponification number shall be determined in accordance with the Tentative Method of Test for Saponification Number of Petroleum Products by Color-Indicator Titration (A.S.T.M. Designation: D 94) of the American Society for Testing Materials.

TENTATIVE REVISIONS OF STANDARDS

Standard Methods of Testing Electrical Porcelain (D 116 - 42):

The following tentative revision outlines the procedure for determining porosity of porcelain by the dye penetration method. The procedure as recommended is in agreement with the method outlined by the American Institute of Electrical Engineers as described in A.I.E.E. Standard for Insulator Tests (No. 41-A).

Section 31.—Add a new Paragraph (*c*) to read as follows:

(*c*) Method C, described in Sections 42 to 46, covers the procedure for determining porosity as indicated by the penetration of an alcohol-dye solution into the porcelain body.

Method C.—Add new Sections 42 to 46, inclusive, comprising the description of the dye penetration method to read as follows, renumbering the subsequent sections accordingly:

Method C. Dye Penetration

42. *Apparatus.*—The apparatus shall consist of a suitable pressure chamber of such dimensions as to accommodate the test specimen when immersed in the dye solution with arrangements for obtaining and maintaining the required pressure for the required time.

43. *Reagent: Fuchsine Solution.*—A solution consisting of 1 g. of basic fuchsine in 1 liter of 50 per cent alcohol is suitable. If a denatured alcohol is used, one should be selected which does not react with the dye to cause fading of the color.

44. *Test Specimens.*—The test specimens shall be freshly broken fragments of the porce-

lain body, having clean and apparently unshattered surfaces exposed. At least 75 per cent of the area of such specimens should be free from glaze or other surface treatment. Fragments approximately ¼ in. in the smallest dimension up to ¾ in. in the largest dimensions are recommended.

45. *Procedure.*—(*a*) The test specimens shall be placed in the pressure chamber and completely immersed in the fuchsine solution.

(*b*) A pressure of approximately 4000 psi. ± 10 per cent shall be applied for approximately 15 hr. or an optional pressure of 10,000 psi. ± 10 per cent for 6 hr. may be used.

(*c*) At the conclusion of the application of test pressure, the specimen shall be removed from the pressure chamber, thoroughly dried, and broken as soon as possible for visual examination.

(*d*) Porosity is indicated by penetration of the dye into the porcelain body to an extent visible to the unaided eye. Penetration into small fissures formed in preparing the test specimen shall be disregarded.

46. *Report.*—The report shall include a statement of the observations recorded in accordance with the examination prescribed in Section 45 (*d*).

Standard Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (D 229 – 42):

The following revisions are intended to replace the present tentative revision[8] submitted in June, 1939. These revisions are proposed in order to bring the conditioning procedure up to date and into conformity with the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 – 41 T):

Section 3.—Change from its present form to read as follows:

3. *Conditioning for Reproducibility.*—The test specimens (Note 1) of flat sheet and plate materials shall be given a conditioning treatment for physical tests (Note 2) in accordance with the Tentative Methods for Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618) of the American Society for Testing Materials, modified as follows:

(*a*) The following materials shall be classified as type *S* (slowly affected by humidity) and conditioned accordingly:

(*1*) Laminated sheets bonded with phenolic, urea, or other thermosetting resins,

(*2*) Methyl methacrylate,

(*3*) Asbestos boards, and

(*4*) Hard rubber.

(*b*) The following shall be classified as type *R* (rapidly affected by humidity):

(*1*) Pressboard and other cellulosic boards, and

(*2*) Cellulosic esters and ethers.

(*c*) Other sheet and plate materials, with the exception of those definitely classified in the individual methods of test or in the specifications for a particular material, shall be classified as prescribed in Methods D 618.

(*d*) *Impact Strength.*—The machined specimens of sheet materials shall be conditioned before testing in accordance with the procedure described for type *S* or type *R* materials, except that the time of conditioning for type *S* materials shall be limited to 48 hr., regardless of the thickness, and that materials may be tested, after cooling in a desiccator, as soon as room temperature has been reached. Tests for type *S* materials shall be made at room temperature maintained at 25 ± 8 C. except where closer limits are specified.

(*e*) *Tensile, Flexural, Compressive, and Bonding Strengths, Rockwell Hardness, and Density.*—For type *S* materials, no conditioning of specimens is necessary except in case of dispute in which case type *S* conditioning procedure shall be followed modified as prescribed in Paragraph (*d*). For type *R* materials the conditioning procedure for that type shall be followed in all cases.

NOTE 1.—Conditioning of specimens may be undertaken: (*1*) for the purpose of bringing the material into equilibrium with normal or average room conditions of 25 C. (77 F.) and 40 per cent relative humidity, (*2*) simply to obtain reproducible results, regardless of previous history of exposure, or (*3*) to subject the material to abnormal conditions of temperature or humidity in order to predict its service behavior.

The conditioning prescribed in these methods to obtain reproducible results may give physical values somewhat higher or somewhat lower than values under equilibrium at normal conditions, depending upon the particular material and test. To insure substantial equilibrium under normal conditions of humidity and temperature, however, will require from 20 to 100 days or more depending upon thickness and type of material and its previous history. Consequently, conditioning for reproducibility must of necessity be used for general purchase specifications and product control tests.

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[8] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 564; also 1941 Supplement, Part III, p. 629.

NOTE 2.—Conditioning of specimens for electrical tests is also necessary to obtain consistent results. The procedure for such conditioning is under consideration by the committee. In order to secure comparative results, specimens should be conditioned at the same temperature and humidity.

Standard Methods of Testing Laminated Tubes Used in Electrical Insulation (D 348 – 42):

This tentative revision consists of the addition of a new method for determining dielectric strength of tubes parallel with the laminations by employing the point to plane procedure.

Section 3.—Add a new Section 3 to read as follows:

3. Dimensional measurements of tubes shall be made in accordance with the Tentative Methods of Measuring Dimensions of Rigid Tubes Used in Electrical Insulation (A.S.T.M. Designation: D 668) of the American Society for Testing Materials.

Sections 15 to 22.—Substitute for these sections new Sections 15 to 21, inclusive, comprising a new procedure for dielectric strength, renumbering the subsequent section accordingly:

15. *Dielectric Strength.*—(*a*) Except as specified below in Sections 16 to 22, the dielectric strength shall be determined in accordance with Tentative Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (A.S.T.M. Designation: D 149) of the American Society for Testing Materials.

(*b*) Tests shall be made transverse or parallel with the wall of the tube, or both, depending upon whether the stress on the tube when in use is to be transverse or parallel with the wall, or both.

16. *Electrodes and Test Specimens:* (*a*) *For Testing in Transverse Direction.*—The inner electrode shall consist of a brass rod, 3 in. in length, with edges rounded to a ¼-in. radius, and of such diameter that it fits snugly inside the tube to be tested. The outer electrode shall consist of a strip of metal foil 2½ in. in width and long enough to extend around the circumference of the tube. The test specimen shall be at least 12 in. in length.

(*b*) *For Testing Parallel with Laminations.*—The test specimens shall be ½ in. in length. A hole shall be drilled into one end of the test specimen in the approximate center of the wall parallel with the major axis of the tube to a depth of ⅜ in. leaving a thickness of ⅛ in. to be tested. A snug-fitting metal-pin electrode, with the end ground to conform with the shape of the drill used, shall be inserted in the hole. The specimen shall be placed on a flat metal plate having a diameter at least ½ in. greater than that of the specimen. This plate shall serve as the lower electrode. Thus, in effect the material shall be tested parallel with lamination in a point-plane gap. The diameter of the hole shall be as shown in the following table:

Nominal Wall Thickness of Tubes, in.	Nominal Hole Diameter for Pin Electrode, in.
$\frac{1}{16}$ to ⅛, excl.	$\frac{1}{32}$
⅛ to ¼, incl.	$\frac{1}{16}$
Over ¼	⅛

NOTE.—For tubes with exceptionally high dielectric strength or for thin-wall tubes, surface flashover may occur. In such cases, specimens shall be drilled to a depth of $\frac{7}{16}$ in. leaving a section $\frac{1}{16}$ in. in thickness to be tested.

17. *Conditioning.*—(*a*) Unless otherwise specified, all test specimens shall be conditioned for 48 hr. at 50 ± 3 C. in a circulating air oven prior to testing. After removal from oven, specimens shall be permitted to cool to room temperature in a desiccator over anhydrous calcium chloride.

(*b*) In the case of tubes to be used at other than room temperature, the dielectric strength characteristics shall be determined over the operating range of temperature. Prior to test, specimens previously conditioned as described in Paragraph (*a*) shall be exposed to each test temperature in a suitable temperature-control chamber for a period in minutes equal to one half the wall thickness of the specimen in mils.

18. *Surrounding Medium.*—The specimens shall be tested, immersed in oil, maintained at the temperature of the test specimen.

19. *Procedure.*—(*a*) Tests shall be made by either the short-time method or the step-by-step method.

(*b*) In tests made by the short-time method, the voltage shall be increased at the rate of 0.5 kv. per sec.

In tests made by the step-by-step method, the voltage at each step shall be applied for 1 min. The voltage shall be increased in increments as follows:

Breakdown Voltage by Short-Time Method	Increment of Increase of Test Voltage
25 kv. or less	1.0 kv.
Over 25 to 50 kv., incl.	2.0 kv.
Over 50 to 100 kv. incl.	5.0 kv.
Over 100 kv.	10.0 kv.

20. *Number of Tests.*—At least five tests shall be made at each temperature in the short-time method, and at least three tests in the step-by-step method. When the range of test temperatures is considerable, tests should be made at not less than five temperatures, if a curve of dielectric strength against temperature is desired.

21. *Report.*—The report shall include the following:

(*1*) A description of the material including name, type, grade, color, size, and name of the manufacturer.

(*2*) A statement of the direction of dielectric stress application whether transverse to or parallel with laminations.

(*3*) The conditioning treatment which the specimens have received.

(*4*) A statement of the procedure used, whether short-time method or step-by-step method.

(*5*) Nominal wall thickness of the tube in inches.

(*6*) The maximum, minimum, and average puncture voltage in kilovolts and volts per mil (Note).

(*7*) Duration of test, if step-by-step method, including the initially applied voltage in kilovolts.

(*8*) The temperature of the test specimen,

(*9*) The size and type of electrodes.

NOTE.—To calculate the volts per mil, the wall thickness in transverse tests on material undisturbed by breakdown but as near the point of breakdown as possible shall be used. For tests parallel with laminations, the thickness of the section shall be measured prior to breakdown. This can be conveniently done by measuring the length of the electrode then the combined length of the specimen and electrode with the electrode inserted in place.

Standard Methods of Testing Laminated Round Rods Used in Electrical Insulation (D 349 – 42):

This tentative revision consists of the addition of a new method for determining dielectric strength of round rods parallel with the laminations by employing the point to plane procedure.

Dielectric Strength Test.—Add new Sections 19 to 25, inclusive, comprising a new procedure for dielectric strength, to read as follows:

19. *Dielectric Strength.*—(*a*) Except as specified below in Sections 20 to 25, the dielectric strength shall be determined in accordance with the Tentative Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (A.S.T.M. Designation: D 149) of the American Society for Testing Materials.

(*b*) Tests shall be made parallel with the major axis of the rod.

20. *Electrodes and Test Specimens.*—The test specimens shall be $\frac{1}{2}$ in. in length. A hole shall be drilled into one end of the test specimen in the approximate center of the rod parallel with the major axis of the rod to a depth of $\frac{3}{8}$ in. leaving a section $\frac{1}{8}$ in. in thickness to be tested. A snug-fitting metal pin electrode with the end ground to conform with the shape of the drill used shall be inserted in the hole. The specimen shall be placed on a flat metal plate having a diameter at least $\frac{1}{2}$ in. greater than that of the specimen. This plate shall serve as the lower electrode. Thus, in effect, the material shall be tested parallel with laminations in a point-plane gap. The diameter of the hole shall be as shown in the following table:

Nominal Diameter of Rod, in.	Nominal Hole Diameter for Pin Electrode, in.
$\frac{1}{8}$ to $\frac{1}{4}$, incl.	$\frac{1}{16}$
Over $\frac{1}{4}$	$\frac{1}{8}$

NOTE.—For rods with exceptionally high dielectric strength or for small diameter rods, surface flashover may occur. In such cases, specimens shall be drilled to a depth of $\frac{7}{16}$ in. leaving a section $\frac{1}{16}$ in. in thickness to be tested.

21. *Conditioning.*—(*a*) Unless otherwise specified, all test specimens shall be conditioned for 48 hr. at 50 ± 3 C. in a circulating air oven prior to testing. After removal from oven, specimens shall be permitted to cool to room temperature in a desiccator over anhydrous calcium chloride.

(*b*) In the case of rods to be used at other than room temperature, the dielectric strength characteristics shall be determined over the operating range of temperature. Prior to test, specimens previously conditioned as described in Paragraph (*a*) shall be exposed to each test temperature in a suitable temperature-control chamber for a period in minutes equal to one half the diameter of the specimen in mils.

22. *Surrounding Medium.*—The specimens shall be tested immersed in oil, maintained at the temperature of the test specimen.

23. *Procedure.*—(*a*) Tests shall be made by either the short-time method or the step-by-step method.

(*b*) In tests made by the short-time method, the voltage shall be increased at the rate of 0.5 kv. per sec.

In tests made by the step-by-step method, the voltage at each step shall be applied for 1 min. The voltage shall be increased in increments as follows:

Breakdown Voltage by Short-Time Method	Increment of Increase of Test Voltage
25 kv. or less	1.0 kv.
Over 25 to 50 kv., incl.	2.0 kv.
Over 50 to 100 kv., incl.	5.0 kv.
Over 100 kv.	10.0 kv.

24. *Number of Tests.*—At least five tests shall be made at each temperature in the short-time method, and at least three tests in the step-by-step method. When the range of test temperatures is considerable, tests should be made at not less than five temperatures, if a curve of dielectric strength against temperature is desired.

25. *Report.*—The report shall include the following:

(*1*) A description of the material including name, type, grade, color, size, and name of the manufacturer.

(*2*) The conditioning treatment which the specimens have received.

(*3*) A statement of the procedure used whether short-time method, or step-by-step method.

(*4*) The maximum, minimum, and average puncture voltage in kilovolts and volts per mil (Note).

(*5*) Duration of test if step-by-step method, including the initially applied voltage in kilovolts.

(*6*) The temperature of the test specimen.

(*7*) The size and type of electrodes.

NOTE.—The thickness of the section shall be measured prior to breakdown. This can be conveniently done by measuring the length of the electrode then the combined length of the specimen and electrode with the electrode inserted in place.

Standard Methods of Testing Shellac Used for Electrical Insulation (D 411 – 42):

This revision is recommended, since the flow of shellac is influenced by the moisture content of the specimen. The test results obtained by conditioning the specimens as recommended are much more consistent than they were formerly. The variation of test results has been reduced from about 25 per cent to 10 per cent.

Section 11 (*a*).—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

(*a*) Each test specimen shall consist of 2 g. of shellac, coarsely ground (approximately *to pass* 20 mesh). [As changing atmospheric conditions have a very marked effect on the flow of shellac, the open test tubes containing the test specimens shall not be exposed to the laboratory atmosphere for more than 24 hr. before testing.] ***The specimens shall be spread out in a shallow vessel and placed in a desiccator over a saturated solution of sodium dichromate with an excess of solid salt and left in this atmosphere (52 per cent relative humidity) at room temperature for at least 24 hr.; the specimens shall be tested immediately upon removal from the desiccator.***

ADOPTION OF TENTATIVE STANDARDS AS STANDARD

Tentative Methods of Test for Thickness of Solid Electrical Insulation (D 374 – 41 T):

The committee recommends that these methods be approved for reference to letter ballot of the Society for adoption as standard without revision.

Tentative Methods of Testing Pin-Type, Lime Glass Insulators (D 468 – 39 T):

The committee recommends that these methods, revised as follows, be approved for reference to letter ballot of the Society for adoption as standard:

Section 5 (*c*).—Change the last two sentences to read as follows by the omission of the words in brackets:

> At least four insulators of each type shall be tested at one time [half without pins and half with standard pins, of the type to be used screwed tightly into the pin holes. The pins shall have well-formed threads, and when threads show wear the pins shall be discarded.]

Section 7.—In order that the description of the application of the load will conform to the American Institute of Electrical Engineers Standard for Insulator Tests (No. 41-A), change the next to the last sentence from its present form: namely

> The load shall be applied uniformly and the speed of the testing machine shall be such that the load can be accurately weighed but shall not exceed 0.25 in. per min. when the machine is running idle.

to read as follows:

> The load shall be started at zero and smoothly brought up in a practically stepless variation to the failure point. The load shall be increased rapidly to approximately 75 per cent of the rated strength of the insulator and at a slower rate from this point to failure. The rate of increase of load from 75 per cent of rating to failure shall be as follows:

Class of Insulator	Type of Test	Increase in Load per Minute in Percentage of Rated Strength	
		Min.	Max.
Pin Type	Cantilever	30	60

Tentative Method of Test for Water Absorption of Plastics (D 570 – 40 T):

Committee D-9 joins with Committee D-20 on Plastics in recommending that this method be adopted as standard, since this test is under the joint jurisdiction of both committees.

ADOPTION OF TENTATIVE REVISIONS OF STANDARDS AS STANDARD

Standard Methods of Testing Electrical Porcelain (D 116 – 39):

The committee recommends that the tentative revision[9] of these methods submitted August, 1940, which relates to the test specimens for water absorption, be referred to letter ballot of the Society for adoption as standard.

Standard Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (D 229 – 39):

The committee recommends that the tentative revisions[8] published in June, 1939, and June, 1940, listing modifications in Sections 16 (*f*) and 38 (*c*) (relating to room temperature), Sections 19 to 22, and 24 to 28 (water absorption), Sections 41 and 42 (Rockwell hardness) with modification in Section 41 as indicated below, and Section 53 (inflammability test) be referred to letter ballot of the Society for adoption as standard:

Section 41.—Change Paragraphs (*a*) and (*b*) from their present form to read as follows:

> 41. (*a*) The procedure described in Standard Method E 18 shall be followed. The adjustment of speed of load application and the time of application of the major load are of great importance. The dash pot on the Rockwell tester shall be so adjusted that the operating handle completes its travel in from 4 to 5 sec. with no specimen on the machine and with the machine set up to apply a major load of 100 kg. When so adjusted, the period taken for mechanism to come to a stop with the specimen in place will vary from about 5 to 15 sec. depending upon particular specimen, the penetrator, and load used. In order to eliminate errors due to "creeping", and to the possibility of weight arm descending far enough to rest on the lifting link or pin (Note) instead of on the specimen, the major load shall be removed from the specimen within 7 to 8 sec. after the handle has been tripped, time being determined by a

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[9] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 569; also 1941 Supplement, Part III, p. 635.

stopwatch. Readings shall be taken on either the M or R scale. The M scale requires a $\frac{1}{4}$-in. ball penetrator, a minor load of 10 kg., a major load of 100 kg., and the reading taken on the red scale. The R scale requires a $\frac{1}{2}$-in. ball penetrator, a minor load of 10 kg., a major load of 60 kg., and readings taken on the red scale. The Rockwell reading shall be taken to the maximum nearest full scale division reached within 45 sec. after removing the major load.

NOTE.—If the arm rests on the link or pin the load or the penetrator or both should be changed.

(*b*) Unless otherwise specified in the individual methods or specifications for a particular material, the choice of scales to be used shall be determined as follows: If readings with the M scale show a total indentation of less than 150 scale divisions upon application of the major load, the M scale shall be used for the Rockwell test. If readings show a total indentation of 150 or more scale divisions the tester shall be changed to the R scale combination. The total indentation shall be determined by the number of divisions the pointer passes through during 7.5 sec. from the time of tripping the handle.

Omit Paragraph (*c*) which reads as follows:

(*c*) For vulcanized fibre, a $\frac{1}{2}$-in. ball penetrator, a minor load of 10 kg., and a major load of 60 kg. shall be used. This combination is known as the R scale. The major load shall be removed within 7 to 8 sec. after the handle has been tripped. Care shall be taken that the weight arm does not descend far enough to rest on the lifting link or pin. If the arm rests on the link or pin, the load or penetrator should be changed.

In Paragraph (*d*), omit the Note which reads as follows:

NOTE.—It should be noted that some materials such as laminated phenolic plates show an appreciable variation in the Rockwell hardness with variation in temperature as small as 8 C. (15 F.).

Standard Methods of Testing Laminated Tubes Used in Electrical Insulation (D 348 – 39):

The committee recommends that the tentative revisions[10] of these methods published in June, 1939, and June, 1940, which pertain to conditioning for reproducibility and the water absorption test, be referred to letter ballot of the Society for adoption as standard.

Standard Methods of Testing Laminated Round Rods Used in Electrical Insulation (D 349 – 39):

The committee recommends that the tentative revisions[11] of these methods published in June, 1939, and June, 1940, which pertain to conditioning for reproducibility, the water absorption test, and the omission of the section on temperature for tensile strength tests, be referred to letter ballot of the Society for adoption as standard.

Standard Methods of Testing Shellac Used for Electrical Insulation (D 411 – 40):

The committee recommends that the tentative revisions[12] of the methods submitted in August, 1941, with changes indicated below, be referred to letter ballot of the Society for adoption as standard. Since the tentative revision was issued, it has been learned that the graduated glass tube prescribed in the revision is a patented proprietary article. Other satisfactory graduated tubes have been found available and for these reasons the recommended change is proposed.

Appendix.—Change Paragraph (*a*) from its present form to read as follows:

(*a*) *Test Tubes.*—Two test tubes, *A*, of the following type should be used:

(*1*) For use with method A, two heat-resistant, graduated glass tubes. These tubes should be 13 cm. in length, 2.5 cm. in outside diameter, and 1.5 mm. in wall thickness. They shall be graduated in 5-mm. divisions beginning 1.1 cm. ($\frac{7}{16}$ in.) from the outside bottom and extending upward to 100 mm. Every 10-mm.

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[10] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 566; also 1941 Supplement, Part III, p. 632.

[11] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 567; also 1941 Supplement, Part III, p. 632.

[12] 1941 Supplement to Book of A.S.T.M. Standards Part III, p. 629.

line shall be numbered. The graduations and numbers shall be in a contrasting color to the background. The colored material used for making these tubes shall be resistant, for a reasonable period of time, to both oil and glycerol at 130 C. and concentrated caustic solutions at 100 C. These tubes are to be used for holding the specimen *S*.

(*2*) For use with method B, plain heat-resistant glass tubes 12.5 cm. in length and 2.5 cm. in outside diameter may be employed.

The test tubes for both methods should be stoppered with tightly fitting corks through which extend small breather tubes, B.

WITHDRAWAL OF STANDARDS

Standard Methods of Testing Molded Materials Used for Electrical Insulation (D 48 – 39):

New tentative methods comprising completely revised procedures for testing molded material were published in August, 1941, and the committee accordingly recommends that these Standard Methods D 48 – 39 be withdrawn, since they are replaced by the new Tentative Methods of Testing Molded Materials Used for Electrical Insulation (D 48 – 41 T).

Standard Methods of Test for Impact Resistance of Electrical Insulating Materials (D 256 – 38):

New tentative methods comprising completely revised procedures and expanded to cover the testing of plastics were published in August, 1941, and the committee accordingly recommends that these Standard Methods D 256 – 38 be withdrawn since they are replaced by the new Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (D 256 – 41 T).

EDITORIAL CHANGES IN STANDARD

Standard Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (D 229 – 39):

Several editorial modifications indicated below in these methods are necessitated by the removal of the test procedures pertaining to vulcanized fibre and their inclusion in new methods of testing vulcanized fibre.[4] The procedures for the determination of silica, zinc chloride, and density are being omitted from Methods D 229 and included in the new tentative methods as well as several references to vulcanized fibre throughout the methods.

Section 1.—Change from its present form to read as follows:

1. These methods cover the procedures for testing stiff, flat sheet and plate materials, such as phenolic and other types of laminated sheets, hard rubber, asbestos composition board, etc., to be used as electrical insulation except as modified by the individual methods or specifications for a particular material.

NOTE.—For tests applying to vulcanized fibre reference should be made to the Tentative Methods of Testing Vulcanized Fibre Used for Electrical Insulation (A.S.T.M. Designation: D 619) of the American Society for Testing Materials.

Section 17.—Omit Note 2 from this section.

Section 40.—Omit the note following this section.

Section 43.—Omit this section which covers the procedure for determining density of vulcanized fibre.

Section 44.—Omit the words "vulcanized fibre" from the heading of this section and change the words "vulcanized fibre" in the text to read "material."

Sections 46 to 49, Inclusive.—Omit these sections covering the procedures for determining zinc chloride and silica in vulcanized fibre.

APPENDIX II

DATA ON POWER FACTOR MEASUREMENTS PARALLEL WITH LAMINATIONS FOR PHENOLIC LAMINATED SHEET

PREPARED BY SECTION O ON LAMINATED PHENOLIC PRODUCTS FOR RADIO, OF SUBCOMMITTEE III ON PLATES, TUBES, AND RODS

For the past four years, Section O has been studying the measurement of power factor parallel with laminations. The parallel plate method was found to give satisfactory results but to be so time-consuming as to make it of little value for either acceptance testing or control.

It was then proposed to use a specimen composed of phenolic laminated strips 5.25 in. in length carefully sawed to a width of 0.250 in. by the thickness, clamped together in a special metal frame. In this test enough strips are used to make a pile-up approximately 5 in. high. This results in a compound sheet specimen approximately 5 by 5 in. square by 0.250 in. thick. Vaseline-coated tin foil electrodes are placed in the center of each face and smoothed carefully into place to eliminate any air pockets. Power factor and dielectric constant measurements are made in accordance with the procedure for a solid sheet of approximately the same size as described in the Tentative Methods of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (A.S.T.M. Designation: D 150) of the American Society for Testing Materials.[1]

Three series of round robin tests were made on various samples using this proposed method. In the first and third series comparative tests were also made using solid samples 6 by 6 in. square by the thickness, with measurements taken perpendicular to the laminations. The following collaborated in this work:

Continental-Diamond Fibre Co.,
National Vulcanized Fibre Co.,
Synthane Corp., and
U. S. Navy Material Laboratory

The data obtained in these round robin tests are presented in Table I. In general, they indicate the following:

1. Where samples are tested under similar conditions, the results of power factor and dielectric constant tests by various laboratories agree within an average deviation of less than plus or minus 5 per cent.
2. Because of much more rapid effect of changes in humidity on strips $\frac{1}{4}$ in. in width necessarily used for parallel power factor measurements than on the 6 by 6 in. samples used for perpendicular measurements, results cannot be expected to be quite as uniform as with the latter method.
3. Whereas the 48-hr. conditioning period at 50 C. gives good results in many cases, it is evidently not long enough to produce the greatest accuracy if the original samples have been exposed too long to high humidity conditions. Average deviations as high as plus or minus 10 per cent have been experienced under these conditions.

The committee will undertake another study of modifying the conditioning procedure in order to further improve the method.

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[1] 1942 Book of A.S.T.M. Standards, Part III, p. 1148.

TABLE I.—RESULTS OF COOPERATIVE TESTS ON POWER FACTOR MEASUREMENTS PARALLEL WITH LAMINATIONS.

First Series (June, 1940)

NOTE.—Samples were N.E.M.A. grade XX. All samples were conditioned for 48 hr. at 50 C. prior to testing. Measurements were made at a frequency of 1 megacycle.

Sample	Direction of Power Factor Test	Value Measured	Synthane	National	Continental	Mean Value	Average Deviation Plus or Minus, per cent
A	Perpendicular	Power Factor	0.0364	0.0364	0.0364	0.0364	0.0
		Dielectric Constant	4.58	4.82	4.93	4.78	2.7
		Loss Factor	0.165	0.175	0.180	0.173	3.3
A	Parallel	Power Factor	0.0559	0.0553	0.583	0.0565	2.1
		Dielectric Constant	5.40	5.73	5.78	5.64	2.8
		Loss Factor	0.302	0.317	0.337	0.319	3.5
B	Parallel	Power Factor	0.0595	0.0655	0.0655	0.0635	4.2
		Dielectric Constant	5.12	5.55	5.41	5.36	3.0
		Loss Factor	0.304	0.364	0.355	0.341	7.3

Second Series (Fall, 1940 and Spring, 1941)[a]

NOTE.—Samples were A.S.T.M. grade 5 (N.E.M.A. grade CE) and were molded under various conditions. A different set of samples but cut from the same sheet was tested by each collaborator. All samples were conditioned for 48 hr. at 50 C. prior to testing. Measurements were made at a frequency of 1 megacycle.

Sample No.	Value Measured	U. S. Navy	Synthane	Mean Value	Average Deviation, Plus or Minus, per cent
I	Power Factor	0.0608	0.0611	0.0610	0.3
	Dielectric Constant	5.30	5.24	5.27	0.6
	Loss Factor	0.322	0.320	0.321	0.3
II	Power Factor	0.0662	0.0682	0.0672	1.5
	Dielectric Constant	5.53	5.42	5.48	1.1
	Loss Factor	0.366	0.370	0.368	0.5
III	Power Factor	0.0592	0.0542	0.0567	4.4
	Dielectric Constant	5.27	5.04	5.16	2.3
	Loss Factor	0.312	0.273	0.293	6.8
IV	Power Factor	0.0665	0.0630	0.0648	2.8
	Dielectric Constant	5.67	5.45	5.56	2.0
	Loss Factor	0.378	0.344	3.61	4.7
V	Power Factor	0.0623	0.0603	0.0613	1.6
	Dielectric Constant	5.50	5.19	5.35	3.0
	Loss Factor	0.343	0.313	0.328	4.6
VI	Power Factor	0.0685	0.0702	0.0694	1.3
	Dielectric Constant	5.71	5.58	5.64	1.2
	Loss Factor	0.392	0.392	0.392	0.0

Third Series (March, 1941)

NOTE.—The same set of samples was tested by each collaborator, and each conditioned the samples for 48 hr. at 50 C. prior to testing. Measurements were made at a frequency of 1 megacycle. The collaborators are listed in order of testing.

Sample No.	Nearest N.E.M.A. Grade	Direction of Power Factor Test	Continental	National	Synthane	Mean Value	Average Deviation, Plus or Minus, per cent
1	P	Perpendicular	0.0385	0.0371	0.0367	0.0374	1.9
		Parallel	0.0518	0.0481	0.0507	0.0502	2.8
2	XXP	Perpendicular	0.0304	0.0294	0.0285	0.0294	2.2
		Parallel	0.0450	0.0459	0.0410	0.0440	4.5
3	XXXP	Perpendicular	0.0281	0.0282	0.0272	0.0278	1.6
		Parallel	0.0489	0.0435	0.0410	0.0445	6.7
4	XXX	Perpendicular	0.0362	0.0363	0.0368	0.0364	0.6
		Parallel	0.0584	0.0533	0.0470	0.0528	7.5
5	CE	Perpendicular	0.0539	0.0456	0.0459	0.0485	7.5
		Parallel	0.0626	0.0512	0.0550	0.0563	7.6
6	P with 1 ply C	Perpendicular	0.0455	0.0400	0.0400	0.0418	5.8
		Parallel	0.0679	0.0565	0.0534	0.0592	9.7

[a] These tests were made in connection with the work of Section N on Product Uniformity, of Subcommittee III.

The results obtained have been consistent enough to warrant approval by Section O and Subcommittee III of the Tentative Method of Test for Power Factor and Dielectric Constant Parallel with Laminations of Laminated Sheet and Plate Insulating Materials[2] which is recommended for approval by Committee D-9 and presentation to the Society for publication as tentative.

Respectfully submitted on behalf of Section O,

G. H. MAINS,
Chairman.

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[2] This method was accepted as tentative by the Society and appears in the 1942 Book of A.S.T.M. Standards, Part III, p. 1179.

REPORT OF SECTIONAL COMMITTEE
ON
ELECTRICAL INSULATING MATERIALS
ASA PROJECT: C59

Sectional Committee C59 on Electrical Insulating Materials held one meeting during the year: in Philadelphia, Pa., on February 17, 1942.

At the February meeting, the present officers of the committee were re-elected for the ensuing term of two years.

The following A.S.T.M. standards, submitted to the American Standards Association on the recommendation of Sectional Committee C59, were approved as American Standard during the year:

A.S.T.M. Standards:

Test for Impact Resistance of Electrical Insulating Materials (A.S.T.M. D 256 - 38; ASA C59.11-1941), and

Specifications for Electrical Gloves (Voltage Rating of Gloves—3000 volts) (D 120 - 40; C59.12-1942).

It is hoped that the following methods, recently submitted to the ASA on the recommendation of the sectional committee, will shortly be accorded approval as American Standard:

A.S.T.M. Standard Methods of Testing:

Sheet and Plate Materials Used in Electrical Insulation (D 229 - 39),

Laminated Tubes Used in Electrical Insulation (D 348 - 39), and

Laminated Round Rods Used in Electrical Insulation (D 349 - 39).

In accordance with action taken at the February meeting, the NEMA Manufactured Electrical Mica Standards, recommended for submittal to the ASA, have been referred to Subcommittee IX on Mica Products of A.S.T.M. Committee D-9 on Electrical Insulating Materials for consideration.

The following NEMA standards, proposed for submittal to the ASA, are still under consideration by a special reviewing committee consisting of W. A. Evans (chairman) and T. Smith Taylor:

NEMA Laminated Phenolic Products Standards, and

NEMA Recommended Practice for Machining and Punching of Laminated Phenolic Plates.

The following A.S.T.M. shellac methods and specifications, recommended by Committee D-1 on Paint, Varnish, Lacquer, and Related Products for submittal to the ASA for approval as American Standard, were reviewed by a special committee of Sectional Committee C59, consisting of J. A. Scott (chairman) and W. A. Evans, to determine whether these standards are acceptable so far as their electrical uses are concerned:

A.S.T.M. Standard Specifications for:

Dry Bleached Shellac (D 207 - 35),

Orange Shellac (D 237 - 41),

Shellac Varnishes (D 360 - 41), and

A.S.T.M. Standard Methods of:

Sampling and Analysis of Shellac (D 29 - 40).

In accordance with the recommendations of this reviewing committee, the sectional committee has referred these standards to Committee D-1 with the suggestion that the scopes of Specifications D 207, D 237, and D 360 be limited to indicate that they do not cover shellac intended for use as an electrical insulation material and that there be a cross reference in Methods D 29 calling attention to the fact that certain additional tests, covered in the Methods of Testing Shellac Used for Electrical Insulation (D 411 – 40), need to be made in the testing of shellac for electrical uses. The methods incorporated in Standard D 411 – 40 are now being considered by a special subcommittee consisting of Dean Harvey (chairman) and R. W. Atkinson.

A preliminary list of available standards for electrical insulating materials has been prepared by the sectional committee which represents the first step in compiling a library of available standards for electrical insulating materials to be deposited in the office of the secretary. This list has yet to be reviewed and any desirable additions or corrections made. If there appear to be duplicate or conflicting standards, it is desirable that the specifications be harmonized and a single standard developed as a National standard. Gaps might also be noted where standards are still needed. It is hoped that this list will be useful as a guide in the activities of the sectional committee.

In view of the shortage of critical materials, much interest was shown at the February meeting in the study of synthetic and new materials. A special group was appointed consisting of F. S. Mapes (chairman), Dean Harvey, and W. A. Evans to give consideration to a broader field of products such as cellulose acetate, polyvinyl chloride, polyvinyl acetate, etc.

The sectional committee now has under consideration the preparation of a handbook covering the properties of insulating materials, particularly electrical properties. It is hoped that this project will be furthered during the year.

Respectfully submitted on behalf of the sectional committee,

H. L. CURTIS,
Chairman.

R. E. HESS,
Secretary.

REPORT OF COMMITTEE D-11
ON
RUBBER PRODUCTS

Committee D-11 on Rubber Products held one meeting during the past year. This was in connection with the spring group meetings of A.S.T.M. committees in Cleveland, Ohio, on March 4, 1942. In addition, the subcommittees have actively carried on their work, and definite progress has been made.

Subsequent to the 1941 annual meeting, Committee D-11 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative Methods of:

Testing Asphalt Composition Battery Containers (D 639 - 41 T), and

Sampling and Testing Rubber Latex (D 640 - 41 T).

Revision of Tentative Methods of:

Chemical Analysis of Rubber Products (D 297 - 40 T).

These recommendations were accepted[1] by Committee E-10 on August 25, 1941, and appear in the 1941 Supplement to the Book of A.S.T.M. Standards, Part III.

The new tentative methods of test for asphalt composition battery containers and for sampling and testing latex both apply in fields where no standardized procedures have hitherto been available, and where such standardization was badly needed. This is particularly true in the case of the asphalt composition battery containers, because of their increasing importance due to the shortage of crude rubber created by the war. The subcommittees have given these methods considerable study and are agreed that they represent the best current practice.

The revisions in the chemical analysis methods simplify and refine the procedures for sample preparation. They also add a new type of extraction apparatus which many laboratories prefer, because the reduced size permits more accurate weighing. In addition, attention is called to possible ignition losses, which had previously been ignored.

During the year, the following standards under the jurisdiction of Committee D-11 were approved as American standard by the American Standards Association, and assigned the ASA numbers indicated:

Standard Methods of:

Sample Preparation for Physical Testing of Rubber Products (D 15 - 41; ASA J1.1-1942),

Tension Testing of Vulcanized Rubber (D 412 - 41; ASA J2.1-1942),

Test for Adhesion of Vulcanized Rubber (Friction Test) (D 413 - 39; ASA J3.1-1942),

Test for Accelerated Aging of Vulcanized Rubber by the Oxygen-Pressure Method (D 572 - 41; ASA J4.1-1942),

Test for Accelerated Aging of Vulcanized Rubber by the Oven Method (D 573 - 41; ASA J5.1-1942), and

Standard Specifications for:

Electrical Gloves (Voltage Rating of Gloves—3000 Volts) (D 120 - 40; ASA C59.12-1942).

Agreement has been reached between Committee D-20 on Plastics and Committee D-11 on Rubber Products, establishing an arbitrary line of demarcation in jurisdiction over rubber-like materials which might be classified either as artificial rubbers or as plastics. It has been agreed that Committee D-11 shall have jurisdiction over products made from the following rubber-like materials, in addition to those made from natural rubber:

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee D-11 reported the following results of the letter ballot vote of a total of 71 ballots returned from a committee membership of 122: D 640, affirmative 48, negative 0, ballots marked "not voting" 23; D 639, affirmative 36, negative 0, ballots marked "not voting" 35; D 297, affirmative 58, negative 0, ballots marked "not voting" 13.

- Butadiene synthetic rubbers:
  - Buna
  - Chemigum
  - Hycar
  - Perbunan
- Butyl Rubber,
- Koroseal,
- Neoprene,
- Polyisobutylene,
- Thiokol, and
- Latices from any of the above materials.

EMERGENCY ALTERNATE PROVISIONS AND EMERGENCY SPECIFICATIONS

During the year Committee D-11 has utilized the new emergency procedure in connection with a number of urgent changes in the specifications for rubber insulated wire and cable. On August 25, 1941, Committee E-10 on Standards accepted for publication in the form of a pink sticker an emergency alternate provision in the Standard Specifications for Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound (D 27 – 41), covering alternate sheetings for use in cable tape, which became necessary owing to the scarcity of materials caused by the war. Subsequently, for similar reasons, additional emergency alternate provisions in these same specifications were recommended and were accepted by Committee E-10 on April 6, 1942. These emergency provisions cover changes in the braid requirements, and changes in the conductor which are necessary because of scarcity of materials. Similar emergency alternate provisions were recommended in Specifications for Performance Rubber Compound (D 353 – 41), for Heat-Resisting Rubber Compound (D 469 – 41), and for Ozone-Resistant Type Insulation (D 574 – 40 T). The test requirements were lowered in the performance compound called for by Specifications D 353 in line with the rubber conservation program. All of these emergency alternate provisions were accepted by Committee E-10 on April 6, 1942. The committee also recommended emergency alternate provisions in the Tentative Specifications for Rubber Sheath Compound (D 532 – 39 T) which were accepted by Committee E-10 on April 6, 1942. These alternate provisions involve some reduction in the quality of the rubber compound, which became necessary in order to save crude rubber in accordance with the present restricted governmental program.

Subsequently, new Emergency Specifications for Rubber Sheath Compound for Electrical Insulated Cords and Cables Where Extreme Abrasion Resistance is not Required, were recommended by the committee and accepted by Committee E-10 on May 8, 1942. They have been published under the A.S.T.M. designation ES – 6. These specifications also are in line with the Governmental program for restricting the use of crude rubber, and covered a lower grade rubber jacket which could be used in many cases in place of the higher quality material specified in Specifications D 532.

The committee wishes to go on record as stating that the new emergency procedure has been found very helpful in its work.

I. NEW TENTATIVE STANDARDS

Tentative Method of Test for Identation of Rubber by Means of the Durometer.[2]—For the past several years Subcommittee XVII on Vibration Absorption, and Technical Committee A on Automotive Rubber have given a great deal of attention to the development of a hardness test instrument for rubber which would be more convenient for rapid inspection work than the instrument prescribed by the A.S.T.M. Method of Test for Hardness of Rubber (D 314 – 39). The Shore durometer is the instrument which has had most wide acceptance, but the committees felt that it was somewhat lacking in reliability and in duplicability of the readings. Many instruments have been investigated, but so far none has been found which possesses all of the characteristics desired. Consequently, the committees have given considerable attention to means whereby the defects of the Shore durometer could be minimized. It is now felt that standardization and calibration of durometers will go a long way toward accomplishing the objective sought. To this end, the committee recommends that the Method of Test for Indentation of Rubber by Means of the Durometer be accepted for publication as tentative, as appended hereto.[2]

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[2] This method was accepted as tentative by the Society and appears in the 1942 Book of A.S.T.M. Standards, Part III, p. 1334.

II. REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends the immediate adoption of revisions in two standard methods of test, as given below, and accordingly requests a nine-tenths affirmative vote at the annual meeting in order that these recommendations may be referred to letter ballot of the Society.

Standard Method of Test for Accelerated Aging of Vulcanized Rubber by the Oxygen-Pressure Method (**D 572 – 41**):[3]

Section 4.—Transfer Paragraphs (*b*) and (*h*) of Section 4 on Apparatus to appear as Paragraphs (*b*) and (*c*) of Section 8 on Procedure for Accelerated Aging, relettering the remaining paragraphs accordingly.

Section 8.—Add the following Note at the end of this section:

NOTE.—For the evaluation of rubber compounds intended for service at elevated temperatures, the above methods may be used with an operating temperature of 80 ± 1 C., employing time intervals as suggested in Section 8 (*b*), or as otherwise agreed upon. It should be noted that by increasing the aging temperature to 80 C. from 70 C. the rate of oxidation may be expected to be approximately double, and if the rubber compound is of a rapid aging type, or if it is contaminated with such materials as copper or manganese, the rate of oxidation may be catalyzed to such extent as to become violent.

Tentative Revision.—Withdraw the tentative revision in Section 4 (*b*) and Section 8 (*b*) submitted in June, 1940.[4]

Standard Method of Test for Accelerated Aging of Vulcanized Rubber by the Oven Method (**D 573 – 41**):[5]

Section 4.—Transfer Paragraph (*c*) of Section 4 on Apparatus to appear as Paragraph (*b*) of Section 8 on Procedure for Accelerated Aging, relettering the remaining paragraphs accordingly.

Section 8.—Add the following Note at the end of this section:

NOTE.—For the evaluation of rubber compounds intended for service at elevated temperatures, the above methods may be used with an operating temperature of 90 ± 1 C., employing time intervals as suggested in Section 8 (*b*), or as otherwise agreed upon. It should be noted that by increasing the aging temperature to 90 C. from 70 C. the rate of oxidation may be expected to be approximately quadruple, and if the rubber compound is of a rapid aging type, or if it is contaminated with such materials as copper or manganese, the rate of oxidation may be catalyzed to such extent as to become violent.

III. ADOPTION OF TENTATIVE STANDARD AS STANDARD

The committee recommends that the Tentative Methods of Test for Viscosity and Total Solids Content of Rubber Cements (D 553 – 39 T)[6] be approved for reference to letter ballot of the Society for adoption as standard without revision. These methods will undoubtedly be added to in the future, but the present procedures have been in the tentative status for three years without adverse criticism, and may be considered as satisfactory for the determinations covered.

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[3] 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 76.

[4] *Ibid.*, p. 635.

[5] *Ibid.*, p. 81

[6] 1939 Book of A.S.T.M. Standards, Part III, p. 928

IV. STANDARDS CONTINUED WITHOUT REVISION

The committee recommends that all standards and tentative standards under its jurisdiction not mentioned in this report be continued in their present status without revision. These have been reviewed and no changes are felt to be justified at present.

The recommendations appearing in this report have been submitted to letter ballot of the committee, which consists of 121 voting members; 62 members returned their ballots, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARD			
Test for Indentation of Rubber by Means of the Durometer........	41	1	20
II. REVISIONS OF STANDARDS, IMMEDIATE ADOPTION			
Test for Accelerated Aging of Vulcanized Rubber by the Oxygen-Pressure Method (D 572 – 41; ASA J 4.1 – 1942)	47	2	13
Test for Accelerated Aging of Vulcanized Rubber by the Oven Method (D 573 – 41; ASA J 5.1 – 1942)..........................	46	2	14
III. ADOPTION OF TENTATIVE STANDARD AS STANDARD			
Tests for Viscosity and Total Solids Content of Rubber Cements (D 553 – 39 T)..................	37	0	25

ACTIVITIES OF SUBCOMMITTEES

During the year there have been several changes in chairmanship of subcommittees: W. F. Tuley resigned as chairman of Subcommittee XVIII on Dynamic Fatigue Testing for Rubber Products and has been succeeded by W. J. Geldard; L. M. Freeman, chairman of Subcommittee XXI on Tests of Liquid Rubber Products has been succeeded by J. F. Anderson; O. M. Hayden has retired as chairman of Subcommittee XIX on Tests for Properties of Rubber and Rubber-like Materials in Liquids, and has been succeeded by J. A. Baty.

Interest in the work of the subcommittees has been well sustained during the year.

Technical Committee A on Automotive Rubber (L. A. Danse, chairman).—Technical Committee A has been very active during the year in the development of standardization and simplification projects on rubber compounds. The first of these projects dealt with the entire range of rubber qualities used in the automotive industry. In the past the various grades supplied by the rubber companies amounted to several hundred. Maintenance of all of these qualities involved substantial waste through stock overage on orders and in connection with the technical task of running them in production. The program and classification finally approved by Technical Committee A reduced the number of standard compounds for the automotive industry to 40, and defines these 40 compounds in terms of their physical properties. It is contemplated that these compounds will answer most of the needs of the entire automotive field. The saving which will be accomplished by adoption of such a standardized compound program is inestimable. The standard list has been submitted to the Society of Automotive Engineers and will also be recommended to the A.S.T.M. for publication.[7] The second project is parallel to the first, but deals with synthetic rubbers instead of natural rubbers. The situation here is somewhat different, in that synthetic rubber is just coming into widespread use, and the standardization program will guide its expansion rather than restrict an already overexpanded field. The synthetic program, also, is nearly ready for publication and will be handled in a manner similar to the natural rubber compound project.

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[7] Published in A.S.T.M. BULLETIN, No. 117, August, 1942, p. 66; also as subsequently revised in compilation of "A.S.T.M. Standards on Rubber Products," February, 1943, issued as separate publication.

Subcommittee I on Hose (E. G. Kimmich, chairman).—This subcommittee has given considerable attention to the question of fire hose. Radical changes have been made in this product by the War Production Board as the result of the need to economize in the use of rubber. The subcommittee is working on this problem and expects, in the near future, to submit to the Society proposed emergency alternate provisions in the Standard Specifications for Cotton Rubber-Lined Fire Hose for Public and Private Fire Department Use (D 296 – 38) to bring them in agreement with the emergency requirements.

This subcommittee has held several meetings apart from the main committee.

Subcommittee V on Insulated Wire and Cable (R. A. Schatzel, chairman).—Following the last annual meeting, Subcommittee V recommended editorial changes in the rubber insulated wire specifications (D 27 – 41), referring to the conductor, intended to bring the specifications into agreement with new specifications adopted by Committee B-1 on Copper and Copper Alloy Wires. These editorial changes were incorporated in the specifications. The subcommittee has been extremely active as is evidenced by the emergency provisions and specifications mentioned earlier in this report.

The subcommittee expects to present revised emergency alternate provisions in the Standard Specifications for Insulated Wire and Cable: Heat-Resisting Rubber Compound (D 469 – 41) through Committee E-10 on Standards later in the year.[8]

Work is in progress on an improved moisture absorption test.

Subcommittee VI on Packings (William Staniar, chairman).—This subcommittee is actively at work on standard methods of test for compressed asbestos sheet which are of great importance in the airplane industry, and expects to recommend them to the Society through Committee E-10 on Standards in the near future.

A section is also working on treated paper. At its March meeting a technical paper was presented by FrederickThorn on "Machines for Testing Reciprocating Packings."[9]

Subcommittee XI on Chemical Analysis of Rubber Products (S. Collier, chairman) organized a section on Methods of Analysis for Synthetic Rubber.

J. A. Baty presented a paper on "A Direct Determination of Rubber by Chromic Acid Oxidation." It is hoped that out of this will come a much needed analytical procedure.

Subcommittee XV on Aging Tests (J. H. Ingmanson, chairman).—This subcommittee has been very active in the investigation of artificial light aging of rubber and has carried out an extensive test program, a summary of which appears in the Appendix to this report.

Subcommittee XIX on Tests for Properties of Rubber and Rubber-Like Materials in Liquids (J. A. Baty, chairman) expects to present through Committee E-10 on Standards[8] a revision in the Tentative Methods of Test for Changes in Properties of Rubber and Rubber-Like Materials in Liquids (D 471 – 40 T) making the so-called Garvey method[10] an alternative to the present method of measuring changes in volume during immersion.

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[8] See Editorial Note, p. 391.

[9] Published in ASTM BULLETIN, No. 116, May, 1942, p. 23.

[10] B. S. Garney, Jr., "An Immersion Test for Rubber Compounds," published in ASTM BULLETIN, No. 109, March, 1941, p. 19.

Subcommittee XXI on Adhesion of Rubber Films (J. F. Anderson, chairman).—This subcommittee is actively working on the problem of adhesion of rubber films, with particular reference to balloon seams.

Comments to date on the methods of sampling and testing rubber latex which were published during the past year have been very favorable.

Committee D-11 has authorized a new subcommittee on Physical Properties of Rubber and Rubber-Like Materials at Subnormal Temperatures.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, J. H. Ingmanson.
Vice-Chairman, S. Collier.
Secretary, Arthur W. Carpenter.

This report has been submitted to letter ballot of the committee, which consists of 121 members; 62 members returned their ballots, of whom 61 have voted affirmatively, and 0 negatively.

Respectfully submitted on behalf of the committee,

O. M. HAYDEN,
Chairman.

ARTHUR W. CARPENTER,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee D-11 presented to the Society through Committee E-10 on Standards proposed Emergency Alternate Provisions in the Standard Specifications for Friction Tape for General Use for Electrical Purposes (D 69 – 38) and for Rubber Insulating Tape (D 119 – 38). These emergency provisions were accepted by Committee E-10 on November 25, 1942, and issued under the designations EA – D 69 and EA – D 119, respectively. They were published in ASTM BULLETIN, No. 120, January, 1943, p. 54, and have been issued in the form of pink stickers for attachment to the standards to which they apply.

Committee D-11 also submitted to Committee E-10 proposed changes in the Emergency Alternate Provisions in the Standard Specifications for Insulated Wire and Cable: Heat-Resisting Rubber Compound (EA – D 469), referred to in this report, which were accepted by Committee E-10 on January 15, 1943. The revised emergency provisions were published in ASTM BULLETIN, No. 120, January, 1943, p. 55, under the designation EA – D 469a, and have been issued in the form of a pink sticker for attachment to the specifications.

On February 18, 1943, Committee E-10 accepted proposed revisions in the Tentative Methods of Test for Changes in Properties of Rubber and Rubber-Like Materials in Liquids (D 471 – 40 T) recommended by Committee D-11. The revised tentative methods appear in the special compilation of "A.S.T.M. Standards on Rubber Products," 1943, p. 131, under the designation D 471 – 43 T, and are also available as a separate reprint.

APPENDIX

DIGEST OF COOPERATIVE TEST PROGRAM ON ACCELERATED LIGHT AGING OF RUBBER

PREPARED BY J. H. INGMANSON[1]

At a meeting held at the time of the 1941 Spring Committee Meeting, Subcommittee XV on Life Tests for Rubber Products of Committee D-11 on Rubber Products decided to conduct a cooperative test to obtain preliminary information with regard to the performance of the Proposed Method of Test for Resistance to Accelerated Light Aging of Rubber Compounds.[2]

While the method as written practically specified the use of the National Carbon Co. 1A Weathering Unit, it was agreed that tests should be made with two other designs of apparatus and that outdoor exposure tests should be made for purposes of correlation. The three types of apparatus used were as follows:

1. "The Eveready Accelerated Weathering Unit 1A" produced by the National Carbon Co. with a flaming arc light source at 60 amp., 50 v. with radiation filtered through Corex D glass, samples at a distance of approximately 18 in. from the light source and subjected to a mild spray of water once every 2 hr.
2. The Weatherometer, type BWMC, produced by the Atlas Electric Devices Co., using a single neutral carbon arc at 17 amp. and 220 v. enclosed in a Pyrex glass bowl with exposed specimens sprayed with water every 20 min.
3. The improved Weatherometer, type DLTS, produced by the Atlas Electric Devices Co., using twin arcs as above and a cycle of 3 hr. light exposure and 1 hr. water spray.

In the course of formulating the test program, it was reported by two laboratories that the rate of deterioration in tensile strength of rubber exposed in the Eveready equipment with Sunshine carbons and Corex glass filters was no more rapid than in direct summer sunlight outdoors, but that without the filters, the rate of deterioration was accelerated by a factor of about ten. It was also reported that unstretched specimens deteriorated as rapidly both on outdoor exposure and in accelerated tests as stretched specimens except as affected by cracking which occurred on stretched samples. Accordingly, both of these facts were considered in the formulation of the program of tests.

Ten laboratories participated in the test program, five laboratories using the Eveready equipment, one the Atlas single arc and one the Atlas twin arc equipment while five laboratories conducted outdoor exposure tests. However, only four of the latter reported test data. The laboratories participating in the tests were as follows:

Atlas Electric Devices Co.
Bell Telephone Laboratories, Inc.
E. I. du Pont de Nemours and Co.
The Firestone Tire and Rubber Co.
The B. F. Goodrich Co.
The Goodyear Tire and Rubber Co.
Manhattan Rubber Manufacturing Division
National Carbon Co.
U. S. Navy, Bureau of Ships
United States Rubber Co.

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[1] Member of the Technical Staff, Bell Telephone Laboratories, Inc., New York, N. Y.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 381 (1940).

The laboratories are designated in the report by letters *A* to *J*. The letter designations, however, do not correspond to the above alphabetical listing.

Five typical rubber compounds were selected for test. These were (1) a tire tread, (2) a white sidewall, (3) a hose cover, (4) a wire insulation, and (5) a neoprene veneer.

Test sheets, 2 by 6 in. in size, of the compounds were prepared. All cures were considered to be technical. While the draft as written requires that test sheets for exposure shall be 0.045 to 0.100 in. thick, preliminary experience had shown that with thinner specimens deterioration was more rapid. Since it was desirable that results be obtained as rapidly as possible, it was agreed that for the test program sheets should be approximately 0.015 in. thick which was considered to be the practicable lower thickness limit.

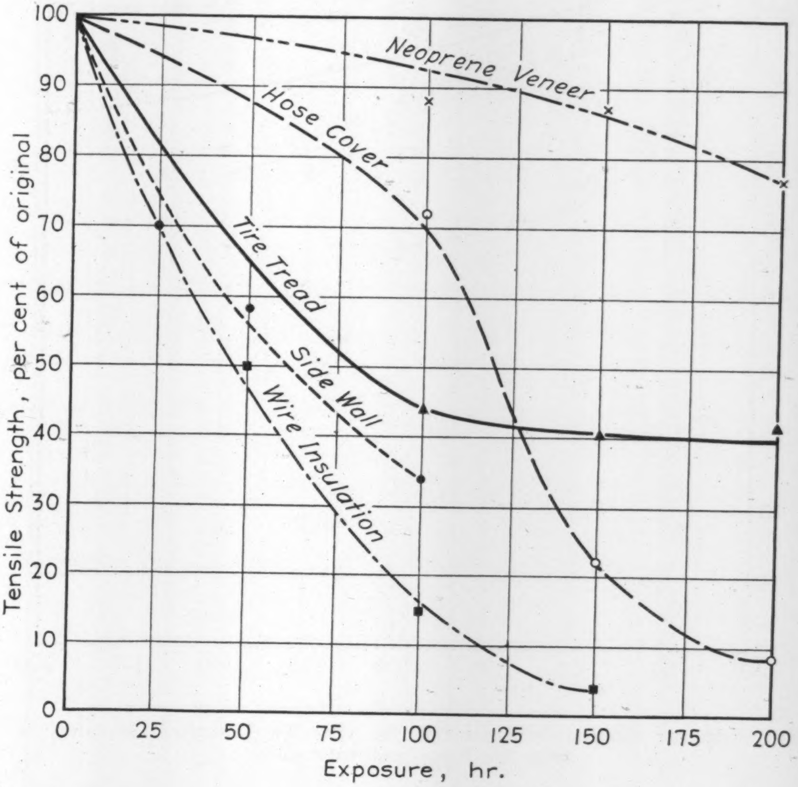

FIG. 1.—Rubber Compounds Exposed in the Eveready Accelerated Weathering Unit 1A. Averaged results from laboratories *A*, *B*, *C*, *D*, and *E*.

The dimensions of the test sheets, 2 by 6 in., permitted cutting 2 tension specimens from each. The A.S.T.M. type A,[3] 0.5-in. die was used for cutting the tension specimens. The program specified that tension tests should be made not less than 24 nor more than 48 hr. after the termination of the exposure. It was impossible to adhere to this schedule in many cases.

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[3] Standard Methods of Tension Testing of Vulcanized Rubber (D 412 – 41), 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 69.

Four sheet specimens 2 by 6 by 0.015 in. of each compound were furnished to each laboratory for exposure in accordance either with the "Aging Machine" or "Outdoor" schedule as given below.

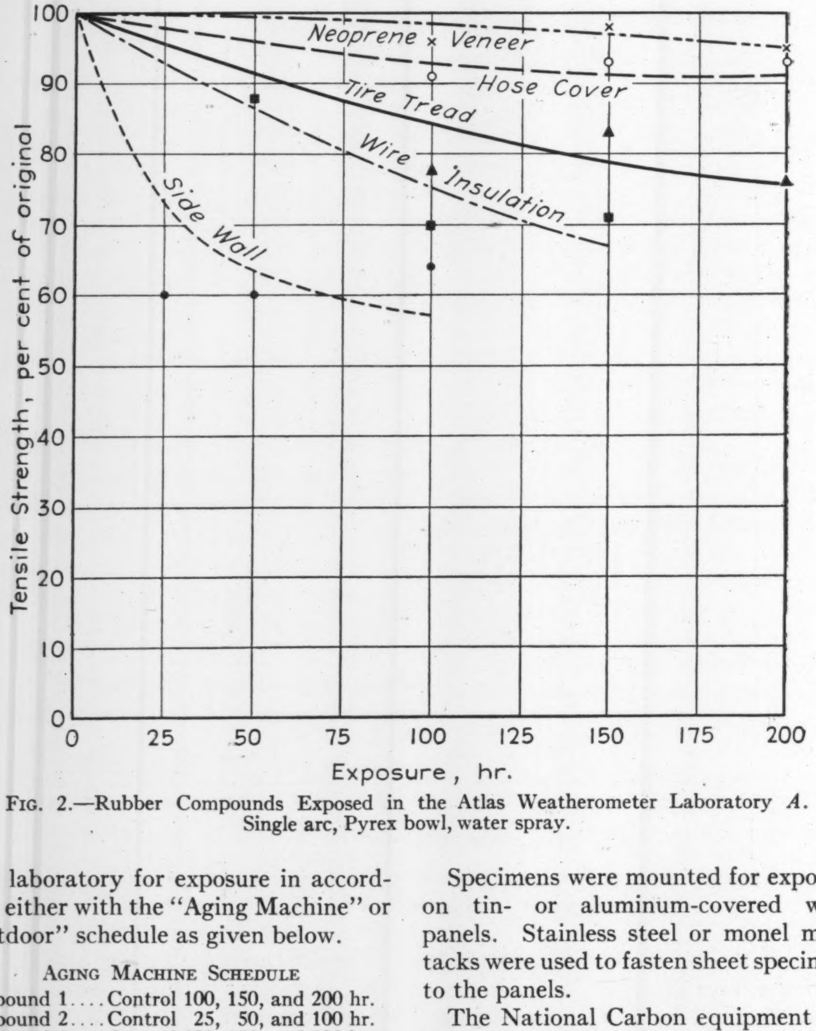

FIG. 2.—Rubber Compounds Exposed in the Atlas Weatherometer Laboratory *A*. Single arc, Pyrex bowl, water spray.

AGING MACHINE SCHEDULE

Compound 1.... Control	100, 150, and 200 hr.
Compound 2.... Control	25, 50, and 100 hr.
Compound 3.... Control	100, 150, and 200 hr.
Compound 4.... Control	50, 100, and 150 hr.
Compound 5.... Control	100, 150, and 200 hr.

OUTDOOR SCHEDULE

Compound 1.... Control	50, 100, and 150 days
Compound 2.... Control	20, 35, and 50 days
Compound 3.... Control	50, 100, and 150 days
Compound 4.... Control	25, 50, and 75 days
Compound 5.... Control	50, 100, and 150 days

Aging tests, both in machines and outdoor, were started late in May, 1941. The longest scheduled outdoor aging period was completed late in October, 1941.

Specimens were mounted for exposure on tin- or aluminum-covered wood panels. Stainless steel or monel metal tacks were used to fasten sheet specimens to the panels.

The National Carbon equipment was operated without filters but with the water spray. The Atlas equipment was used with the water spray but not the freezing cycle. Outdoor exposures were at 45 deg. to the horizontal facing south.

Reports of light intensities for Eveready equipment used by three laboratories as determined by the A.S.T.M. Proposed Method for Calibrating a Light Source Used for Accelerating the Deterioration of Rubber[4] using the decomposition of uranyl oxalate are given below. Ambient air temperature at the plane of the samples as reported is also given.

Laboratory	Light Intensity, mg. of oxalic acid	Ambient Temperature, deg. Fahr.
A	5.6	112
B	...	130
C	5.0	131 to 145
E	6.5	...

The averaged results of tension tests for each compound from the five laboratories using Eveready equipment are plotted in Fig. 1. Figures 2 and 3 show the results of tension tests on all five compounds aged in the Atlas single arc and Atlas twin arc machine, respectively. Figure 4 shows the average of outdoor, direct sunlight aging for the five compounds.

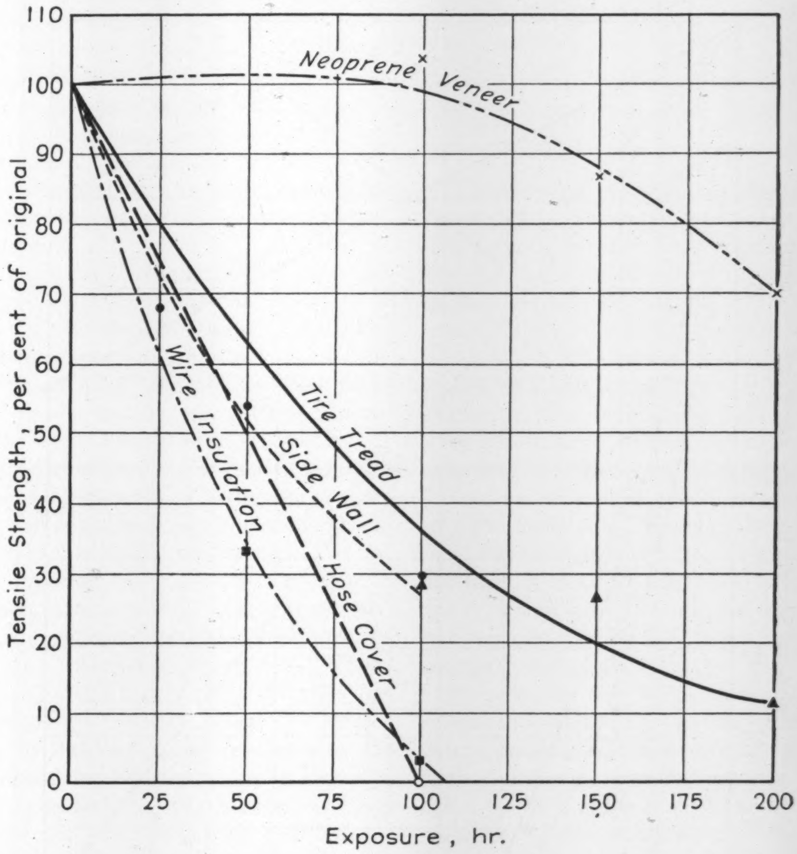

FIG. 3.—Rubber Compounds Exposed in the New Atlas Weatherometer Laboratory *F*. Twin arc, Pyrex globe, water spray.

DISCUSSION

The final tensile strength results both in the light aging machines and in direct sunlight show the neoprene veneer stock to be most resistant to deterioration. In all cases the same order of evaluation was obtained in the Eveready equipment as in outdoor aging. The order of evaluation by the various test methods follows:

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[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 385 (1940).

Outdoor	*Eveready*	*Twin Arc*	*Single Arc*
Neoprene	Neoprene	Neoprene	Neoprene
Tire tread	Tire tread	Tire tread	Hose cover
Hose cover	Hose	Sidewall	Tire tread
Sidewall	Sidewall	Hose cover	Sidewall
Insulation	Insulation	Insulation	Insulation

Among the various laboratories using the Eveready equipment, laboratory *A* was most severe with each of the five compounds. In the case of the tire tread compound, laboratory *A* showed a final deterioration of about 80 per cent. Deterioration with the same equipment in the other four laboratories varied from about 50 to 63 per cent. In the case of the white sidewall compound, laboratory *A* showed a deterioration of over 90 per

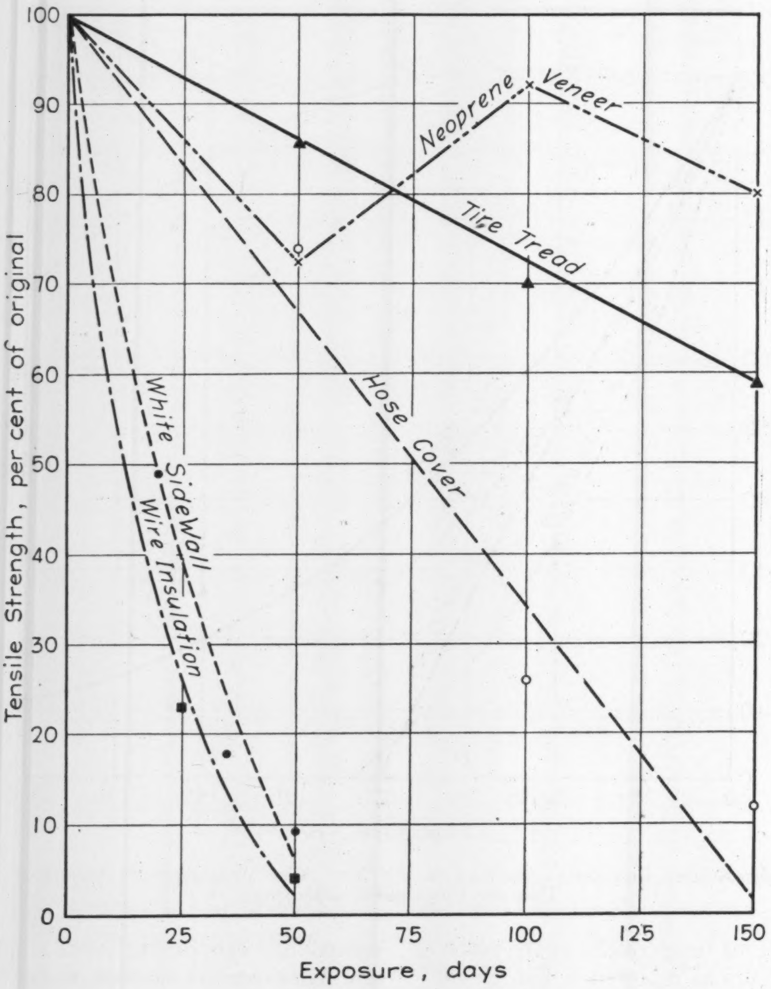

FIG. 4.—Outdoor Exposures, May to October, 1941. Average of laboratories *A*, *G*, and *H*.

cent, laboratory *B* almost 80 per cent, and the other three laboratories between 50 and 60 per cent. In the case of the hose cover compound, laboratories *B*, *D*, and *E* reported results reasonably close together with laboratory *A* results most severe and laboratory *C* reported intermediate results which were less severe but quite well in line finally. All test results in the case of the wire insulation compound showed deterioration of over 90 per cent. A variation of 15 to 37 per cent deterioration was reported from the five laboratories for the Neoprene veneer compound.

Among the four laboratories reporting outdoor exposure tests the variation in final deterioration for the white sidewall and insulation compound were quite consistent. In the case of the other three compounds, the results of three laboratories were consistent, laboratory *A* being more severe in the case of the hose cover and laboratory *J* being more severe with the tire tread and Neoprene stock.

A possible cause for variation in results reported for Eveready exposure is temperature variation. Only three laboratories reported ambient temperatures adjacent to exposed specimens. These varied from 112 to 145 F., the latter being reported by laboratory *C*. Variations in outdoor aging results may be accounted for by variations in locality, local temperature, prevalence of dust, rainfall and other climatic conditions.

The single arc Weatherometer showed little deterioration as compared with the other exposures. The twin arc Weatherometer gave results which were somewhat more severe than the average of those obtained by the five laboratories using the Eveready equipment.

The degree of deterioration obtained in the Atlas twin arc Weatherometer and the average for the Eveready as compared with the average deterioration obtained on outdoor exposure varied considerably for the five compounds tested. Approximate exposure time in the machines in hours equivalent to 1-day outdoor exposure are given below.

Compound	Eveready	Atlas Twin Arc
Tire tread	1.5	1.0
White sidewall	2.7	2.5
Hose cover	1.4	0.6
Insulation	3.0	1.9
Neoprene	2.5	1.9
Average	2.2	1.6

Visual examination of weathered specimens clearly demonstrated differences in character of the weathered surface layer not only between the naturally and artificially weathered samples but (to a lesser degree) between the weathering units of the different laboratories. This indicates a lack of uniformity in performance of the weathering units, which may result from differences in location resulting in differences in temperature, from differences in character and volume of water supply, in the cycle, the position of the sample in the unit, which affects the way in which the sample is hit by the water spray and the volume of water passing over its surface, the manner of support, and so on.

Removal of the filters unfortunately results in a disturbance of the ventilation conditions around the arc and an increase in the temperature at the sample. Considerable heat aging must have occurred which is not differentiated from the light aging. That the temperature of exposure was too high is indicated by the fact that some of the samples—particularly compound 3—became tacky on the side remote from the light source.

While the appearance of the surfaces was of the same general character in the specimens weathered in the Eveready unit, it was definitely different from that of the outdoor specimens. In the case of compounds 1 and 3, for example, the surface skin tended to bend down into the cracks in the outdoor samples, while the Eveready unit samples produced patches of skin with a tendency to curled-up edges giving the appearance of flaking. The Weatherometer samples had, in general, a finer textured skin than the others.

From these observations it would appear that a problem to be solved is to secure uniformity in performance between identical units operated in different laboratories. To this end it would seem advisable to make a much more detailed study of a single compound to establish the reasons for departure from such uniformity.

CONCLUSION

The Eveready unit without filters and the Atlas Twin arc Weatherometer effect a more rapid deterioration of vulcanized rubber on exposure than outdoor exposure in the summer. The degree of acceleration varies with the particular compound being tested. In these tests it was found that from 0.6 to 3.0-hr. exposure in the machines, depending on the compound, was equivalent to 1 day of outdoor summer exposure. The procedure followed in making Eveready tests is not sufficiently standardized to obtain satisfactorily comparable results in several laboratories with a wide variety of compounds. The Atlas single arc equipment shows little acceleration in aging rate as compared with the Atlas twin arc and Eveready equipment as used in these tests. No broad conclusions can be drawn from the results of tests in the Atlas twin arc equipment since results were reported from only one of these machines. It is suggested that measurements of the total radiation may be of greater significance than measurements in the ultraviolet region alone. Further standardization in connection with ventilation and maintenance of a constant uniform temperature around the exposed specimens appears desirable. Further work is required to determine the effect of thickness of exposed sample on rate of deterioration. If exposure periods are to be for time periods such as were used in these tests it may be desirable to use thicker specimens.

REPORT OF COMMITTEE D-12
ON
SOAPS AND OTHER DETERGENTS

Committee D-12 on Soaps and Other Detergents held one meeting this year on March 9 and 10, 1942, at the Hotel New Yorker, New York, N. Y. There was a very large attendance, over two-thirds of the membership being present.

Mr. R. E. Divine, after 52 years in the soap industry, retired this year. He will be greatly missed at committee meetings, because of his invaluable experience. He has been replaced by Burr H. Ritter as representative of the Hooker Electrochemical Co.; C. C. Ziegler replaces Mr. Divine on the Advisory Committee. W. H. Koch has replaced J. E. Doherty on the Advisory Committee.

The following additional changes in personnel of the committee have occurred:

New Members:

G. D. Marrocco, representing Carman and Co.
Earl D. McLeod, representing Arnold Hoffman and Co.
Captain Ross A. Wright, Quartermaster Corps, Laundry Branch, War Department.
Director, U. S. Naval Engineering Station, Annapolis, Md.

New Representatives:

Ralph S. Frazer replacing L. T. Howells, Cowles Detergent Co.
L. B. Parsons replacing J. E. Doherty, Lever Brothers.
G. A. Wright replacing J. A. Jennaman, E. I. du Pont de Nemours & Co., Inc.

Resigned from Society:

J. A. Winkler, Consulting Textile and Chemical Engineer.

C. B. Hogaboom has been designated as the representative of Committee B-8 on Electrodeposited Coatings on Committee D-12.

The present membership of the committee is 71, of whom 35 are classified as producers, 19 as consumers, and 17 as general interest members.

The election of officers for the ensuing term of two years resulted in the reelection of the present incumbents.

At its March meeting, the committee devoted much of its time to studying emergency provisions in the specifications under its jurisdiction necessitated by shortages of materials. This study was particularly appropriate, since the War Production Board was preparing regulations controlling the use of fat stocks and placing limits on the glycerol content of soaps in order to conserve the glycerol. The proposals of the W. P. B. were to place a limit of 0.7 per cent on glycerol in boiled soaps, but to allow 2.75 per cent content in cold-made and semiboiled soaps when plants are small and have no glycerol recovery facilities. Attention of the manufacturers of soaps is called to the fact that soaps must be made in accordance with the regulations of the W. P. B. for the duration of the National Emergency. The Emergency Alternate Provisions in several of the specifications for soap and soap products are listed later in this report.

I. NEW TENTATIVE STANDARDS

The committee recommends that the following three new specifications be accepted for publication as tentative:[1]

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[1] These specifications were accepted as tentative by the Society and appear in the 1942 Book of A.S.T.M. Standards, Part III, p. 1529, 1533, 1531, respectively.

Tentative Specifications for:

Compound Chip Soap (with Rosin),
Compound Powdered Soap (Granulated, with Rosin), and
Built Soap, Powdered (Type D) (D 533 - 42 T), to be added, when adopted, to the Standard Specifications for Built Soap, Powdered (D 533 - 41).[2]

II. REVISIONS OF TENTATIVE STANDARDS

The committee recommends that the following tentative standards be revised as indicated below and continued as tentative:

Tentative Specifications for Tetrasodium Pyrophosphate (Anhydrous) **(D 595 - 40 T)**,[3] revised as follows:

Section 3 .—Add a footnote reference letter "*a*" after the requirement for 97.5 per cent tetrasodium pyrophosphate in the table of chemical composition requirements and add a new footnote to read as follows:

[a] When the sample is taken at other than the place of manufacture, it shall be recognized that tetrasodium pyrophosphate (anhydrous) is slightly hygroscopic. In the case of shipments in which the tetrasodium pyrophosphate content is less than 97.5 per cent, reference should be made to Section 5.

Section 4 .—Change from its present form: namely,

4. Tetrasodium pyrophosphate (anhydrous) shall be purchased by net weight.

to read as follows:

4. *Basis of Purchase.* — (*a*) Tetrasodium pyrophosphate (anhydrous) shall be purchased by net weight. When, however, due to hygroscopicity the tetrasodium pyrophosphate content is less than 97.5 per cent, a corresponding gain in weight of the package shall be expected, and the net weight shall be based on a product containing 97.5 per cent of tetrasodium pyrophosphate. The net weight of the material to be paid for shall be calculated as follows:

$$W = \frac{R \times T}{97.5}$$

where:

W = weight of the material to be paid for,
R = net weight of material as received, and
T = total tetrasodium pyrophosphate calculated as percentage of $Na_4P_2O_7$.

(*b*) In the event that the calculation under Paragraph (*a*) results in a figure greater than 100 per cent, the seller waives the right to invoice the purchaser for any amount in excess of 100 per cent.

New Section.—Add a new Section 5 on rejection to read as follows:

5. *Rejection.*—Material which has picked up sufficient water to reduce the tetrasodium pyrophosphate content to 95 per cent or lower, may be rejected at the option of the purchaser.

New Section.—Add a new Section 6 to read as follows to replace the present note at the end of these specifications. This editorial change introduces a reference to the methods for sampling and chemical analysis of tetrasodium pyrophosphate completed in 1941.[4]

6. *Methods of Sampling and Analysis.*—The material shall be sampled and analyzed in accordance with the Tentative Methods of Sampling and Chemical Analysis of Special Detergents (A.S.T.M. Designation: D 501) of the American Society for Testing Materials.[5]

Tentative Definitions of Terms Relating to Soaps and Other Detergents **(D 459 - 41 T)**,[6] revised as follows:

Change the definition for detergent to read as follows by the addition of the italicized words and the omission of the word in brackets:

Detergent.—Any [material] *compound or mixture* which cleans.

Add the following new definitions:

Detergency.—Effectiveness of soil removal.

Drycleaning Soap (*Drycleaning Aid*).—Any compound or mixture added to a drycleaning solvent to increase cleaning effectiveness.

Wet-Cleaning (*in Drycleaning Industry*).—Special cleaning processes with aqueous media, employed in the drycleaning industry.

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[2] 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 197.

[3] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 515.

[4] *Proceedings*, Am. Soc. Testing Mats, Vol. 41, p. 433 (1941).

[5] 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 611.

[6] *Ibid.*, p. 617.

III. Revisions of Standards, Immediate Adoption

The committee recommends for immediate adoption revisions in following standards, and accordingly asks for a favorable nine-tenths vote at the annual meeting in order that these modifications may be referred to letter ballot of the Society:

Standard Specifications for Alkaline Soap Powder **(D 534 – 41)**[7] the changes recommended are largely editorial:

Section 1 .—Change to read as follows by the addition of the italicized word:

> 1. These specifications cover *alkaline* soap powder.

Section 2 .—Change the first sentence to read as follows by the omission of the words in brackets:

> Soap powder shall be a uniform mixture of soap and [sodium carbonate, with or without other] alkaline salts, in powdered form.

Section 3 .—In the table of chemical composition requirements add the word "calculated" after the words "total alkalinity."

Standard Methods of Sampling and Chemical Analysis of Soaps and Soap Products **(D 460 – 41):**[8]

Section 59 .—Delete the first sentence of the note preceding this section which read as follows:

> Since no available methods are known for determining sodium ion, it is necessary to determine this by difference.

Such methods are known, but the committee feels that the determination by difference is still the best method.

IV. Tentative Revision of Standard

The committee recommends that the following revisions in a standard method be accepted for publication as tentative:

Standard Methods of Sampling and Chemical Analysis of Soaps and Soap Products **(D 460 – 41):**[8]

New Sections.—Add the following description of the procedure for determining tetrasodium pyrophosphate ($Na_4P_2O_7$) in soaps and soap products as new Sections 49 and 50 renumbering the subsequent sections accordingly.

49. *Reagents:* (*a*) *Neutral Ethyl Alcohol* (*95 per cent*).—Denatured ethyl alcohol, 95 per cent. neutral to phenolphthalein, either Formula No, 3-A or No. 30 of the U. S. Internal Revenue Bureau.

(*b*) *Hydrochloric Acid* (*0.50 N*).

(*c*) *Sulfuric Acid* (*0.1 N*).

(*d*) *Zinc Sulfate Solution* (*7 per cent*).—Dissolve 125 g. of c.p. $ZnSO_4 \cdot 7H_2O$ in water and dilute to 1 liter, filter, and adjust the pH to exactly 3.8 with a glass electrode using 0.1 *N* H_2SO_4.

(*e*) *Sodium Hydroxide* (*0.5 N*).—Standardize the 0.5 *N* NaOH against c.p. $Na_4P_2O_7$ prepared by recrystallizing the technical product three times from water, and igniting at 400 C. to constant weight.

50. (*a*) *Procedure.*—Accurately weigh a sample containing an equivalent of approximately 1 g. of $Na_4P_2O_7$, and digest with 200 ml. of hot, freshly boiled, neutral alcohol. Filter through a filter paper or Gooch crucible with suction, and wash with hot alcohol until free from soap. Change receivers, and wash the alcohol-insoluble portion remaining on the filter paper with hot water until washings are neutral to phenolphthalein. Transfer to the filtrate a suitable vessel, cool, and add sufficient distilled water so that the resulting solution will just cover the electrodes of a glass electrode titration assembly. Adjust the pH of the solution to exactly 3.8 with 0.5 *N* HCl. Add 50 ml. of zinc sulfate solution (7 per cent), and allow 5 min. for the reaction to become complete as shown by the pH becoming constant. Titrate the liberated acid with 0.5 *N* NaOH until a pH of 3.8 is again reached. This titration is a measure of the pyrophosphate content.

(*b*) *Calculations.*—Calculate the percentage of tetrasodium pyrophosphate in the soap as follows:

$$Na_4P_2O_7\text{, per cent} = \frac{\text{ml. of NaOH} \times F}{\text{wt. of sample}} \times 100$$

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[7] 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 199.

[8] *Ibid.*, p. 201.

where F = grams of $Na_4P_2O_7$ equivalent to 1 ml. of the NaOH solution used for the titration as calculated by standardization against c.p. $Na_4P_2O_7$.

V. ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the following tentative standards be approved for reference to letter ballot of the Society for adoption as standard:

Tentative Specifications for:

Olive Oil Solid Soap (Type A, Pure; Type B, Blended) (D 592 – 41 T),[9]
Olive Oil Chip Soap (Type A, Pure; Type B, Blended) (D 630 – 41 T),[9]
Palm Oil Chip Soap (Type A, Pure; Type B, Blended) (D 536 – 41 T),[9]
Salt-Water Soap (D 593 – 40 T),[10] and

Tentative Method of Test for:

Carbonates as Carbon Dioxide in Soaps and Soap Products (Evolution-Volumetric Method (D 460 – 41 T)[11] to be added to Standard Methods D 460 – 41.[8]

VI. EMERGENCY ALTERNATE PROVISIONS

As mentioned earlier in the report, the committee has recommended Emergency Alternate Provisions in a number of its soap specifications for the duration of the emergency. The standards and tentative standards affected are as follows:

Standard Specifications for:

Milled Toilet Soap (D 455 – 39),[12]
Chip Soap (D 496 – 39),[12]
Powdered Soap (Nonalkaline Soap Powder) (D 498 – 39),[12]
White Floating Toilet Soap (D 499 – 39),[12]
Built Soap, Powdered (D 533 – 41),[2]
Palm Oil Solid Soap (Type A, Pure; Type B, Blended) (D 535 – 41),[13]

Tentative Specifications for:

Palm Oil Chip Soap (Type A, Pure; Type B, Blended) (D 536 – 41 T),[9]
Olive Oil Solid Soap (Type A, Pure; **Type B,** Blended) (D 592 – 41 T),[9]
Salt-Water Soap (D 593 – 40 T),[10] and
Olive Oil Chip Soap (Type A, Pure; Type B, Blended) (D 630 – 41 T).[9]

These Emergency Alternate Provisions with the exception of EA – D 496 and EA – D 498 were accepted by Committee E-10 on Standards, April 6, 1942, and have been published as stickers for attachment to the specifications to which they apply. They have also been published in ASTM BULLETIN No. 116, May, 1941.[14]

The Emergency Alternate Provisions in Specifications D 496 and D 498 were accepted by Committee E-10 on May 15, 1942, and have been published as stickers for attachment to the specifications to which they apply.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 71 members; 45 members returned their ballots, with the results shown in Table I.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Methods of Testing (J. B. Crowe, chairman):

Section A on Soaps (M. L. Sheely, chairman) has recommended that the Tentative Method of Test for Carbonates as Carbon Dioxide in Soaps and Soap Products (Evolution-Volumetric Method) (D 460 – 41 T) be adopted as standard and incorporated in the Standard Methods of Sampling and Chemical Analysis of Soaps and Soap Products (D 460 – 41). The subcommittee also completed a new procedure for determining tetrasodium pyrophosphate in soaps and soap products which is being submitted for publication as a tentative revision of Standard Methods D 460 – 41, as indicated earlier in the report.

Results obtained with this procedure

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[9] 1941 Supplement to Book of A.S.T.M. Standards, Part III, pp. 602, 604, 606.

[10] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p 512.

[11] 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 608.

[12] 1939 Book of A.S.T.M. Standard, Part III, pp. 483, 479, 481, 485.

[13] 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 195.

[14] See also Editorial Note, p. 406.

in cooperative tests by 11 laboratories are presented in Table II.

This subcommittee is studying the determination of free alkali and carbonates in potash soaps, particularly the use of *iso*propyl alcohol instead of ethyl alcohol, and while the method shows promise, further work is necessary. The present method is not too accurate and the subcommittee hopes that the procedure under consideration will prove more satisfactory.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Spec. for Compound Chip Soap (with Rosin)	42	0	3
Spec. for Compound Powdered Soap (Granulated, with Rosin)	42	0	3
Spec. for Built Soap, Powdered (Type D) (D 533 - — T)	43	0	2
II. REVISIONS OF TENTATIVE STANDARDS			
Spec. for Tetrasodium Pyrophosphate (Anhydrous) (D 595 - 40 T)	43	0	2
Def. of Terms Relating to Soaps and Other Detergents (D 459 - 41 T)	43	0	2
III. REVISIONS OF STANDARDS, IMMEDIATE ADOPTION			
Spec. for Alkaline Soap Powder (D 534 - 41)	42	0	3
Methods of Sampling and Chemical Analysis of Soaps and Soap Products (D 460 - 41)	42	0	3
IV. TENTATIVE REVISION OF STANDARD			
Methods of Sampling and Chemical Analysis of Soaps and Soap Products (D 460 - 41)	41	0	4
V. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Spec. for Olive Oil Solid Soap (Type A, Pure; Type B, Blended) (D 592 - 41 T)	42	0	3
Spec. for Olive Oil Chip Soap (Type A, Pure; Type B, Blended) (D 630 - 41 T)	42	0	3
Spec. for Palm Oil Chip Soap (Type A, Pure; Type B, Blended) (D 536 - 41 T)	41	0	4
Spec. for Salt-Water Soap (D 593 - 40 T)	43	0	2
Test for Carbonates as Carbon Dioxide in Soaps and Soap Products (Evolution - Volumetric Method) (D 460 - 41 T)	43	0	2

Section B on Sulfonated Detergents (Ralph Hart, chairman) is studying a method for determining water-immiscible organic solvents, volatile with steam in sulfonated (sulfated) oils and hopes to have a report ready for the next meeting.

Section C of Subcommittee I and Section D of Subcommittee II on Dry Cleaning Detergents (G. P. Fulton, chairman) reports progress on a most difficult problem. These sections are studying a method for evaluating dry-cleaning agents by actual tests, but are confronted by the problem of finding a means of producing soiled pieces that will satisfy the following requirements: (*a*) The type of soil should be similar to actual soil as it is found on garments, and the grease or oil incorporated with the soil should have, in general, the same solubility characteristics in dry-cleaning solvent as has the grease or oil on actual garments, (*b*) The method

TABLE II.—RESULTS OF COOPERATIVE TESTS FOR DETERMINATION OF TETRASODIUM PYROPHOSPHATE ($Na_4P_2O_7$) IN SOAP.

Laboratory		Moisture, per cent	$Na_4P_2O_7$ in Soap as Received, per cent	$Na_4P_2O_7$, calculated on 5 per cent moisture basis, per cent
Armour and Co.		3.83	12.30	12.15
		3.81	12.39	12.24
	Avg.	3.82	12.35	12.20
National Bureau of Standards		3.71	.....	12.00
Fels and Co.		3.46	12.05	11.86
Foster D. Snell, Inc.		.....	.....	11.94
Hercules Experiment Station, Hercules Powder Co.		3.4	11.9	11.69
		3.4	11.9	11.69
	Avg.	3.4	11.9	11.69
Hooker Electrochemical Co.		3.63	11.99	11.83
Lever Brothers Co.		3.59	12.20	12.02
Los Angeles Soap Co.		3.31	12.19	11.98
		3.41	12.11	11.90
	Avg.	3.36	12.15	11.94
Proctor & Gamble Co.		2.96	12.02[a]	11.77
Swift and Co.		3.15	.....	11.90
Allen B. Wrisley Distributing Co.		3.71	12.22	12.05
Grand Average		.....	.....	11.93
High		.....	.....	12.20
Low		.....	.....	11.69

[a] Solution of NaOH (0.5 *N*) was standardized against constant boiling HCl instead of $Na_4P_2O_7$.

of application of the soil should be similar to the actual contacting of soil by garments, (*c*) The fabric in the soiled pieces should be similar to the material in the garments which are to be drycleaned. Several investigators who have made such soiled pieces have been asked for opinions as to what factors should be controlled during the soiling of fabrics in order to obtain typical soil. The National Association of Dyers and Cleaners has prepared some soiled pieces for drycleaning; the results of this work are to be considered by these sections for comment and suggestions looking toward the development of suitable specimens.

Section D on Special Detergents (W. H. Koch, chairman) will study the determination of the loss on ignition and of the water content of tetrasodium pyrophosphate during the coming year.

Subcommittee II on Specifications (F. W. Smither, chairman):

This subcommittee held one meeting during the year, and its various sections have been active.

Section A on Textile Soaps (C. A. Nutting, chairman) and Section C on Straight Soaps (Israel Katz, chairman) have continued working jointly. These sections have recommended that the following tentative specifications be adopted as standard: Tentative Specifications for Olive Oil Solid Soap (Type A, Pure; Type B, Blended) (D 592 – 41 T), Olive Oil Chip Soap (Type A, Pure; Type B, Blended) (D 630 – 41 T), and Palm Oil Chip Soap (Type A, Pure; Type B, Blended) (D 536 – 41 T).

These sections also completed the new Proposed Tentative Specifications for Compound Chip Soap (With Rosin) and for Compound Powdered Soap (Granulated, with Rosin) mentioned earlier in this report. Due to shortages developing in various oil and fat stocks, it was thought essential that these specifications should be promulgate at this time to allow their use in additio to the use of Standard Specifications fo Chip Soap (D 496 – 39) and for Powdered Soap (Nonalkaline Soap Powder) (D 498 – 39). They are recommended for publication as tentative rather than as Emergency Standards, since it is expected that they will be valuable for use after the duration of the war.

In addition, these sections have developed Emergency Alternate Provisions in several of the soap specifications as indicated earlier in the report. The War Production Board is considering regulations on the amount of glycerol to be left in soap, and the limitation of the use of coconut, palm kernel, and babassu oils in all products and will doubtless issue further limitations in the future.

These sections also plan to study liquid soaps during the coming year.

Section B on Built Soaps (Fred Krassner, chairman) has proposed Emergency Alternate Provisions in the Specifications for Salt-Water Soap (D 593 – 40 T) in order to provide for the development of products made from other than coconut oil which is no longer available, and also with a view to producing a soap acceptable under these specifications. The section has also recommended for publication as tentative a type D built powdered soap to be added, when adopted, to the Standard Specifications for Built Soap, Powdered (D 533 – 41) as given earlier in the report. The new soap is essentially the same as the present three types except that it contains no requirement for titer, and the allowable rosin content is 10 per cent. Type D soap has also been proposed as Emergency Alternate to be substituted for types A, B, or C in Standard Specifications D 533 – 41. This section has also recommended for immediate adoption, revisions in the Standard Specifications for Alkaline Soap Powder (D 534 – 41) as well as some minor editorial changes.

Consideration has been given to specifications for scouring powders but due to the results of some recent studies on abrasives the writing of these specifications has been postponed.

Section E on Sulfonated Detergents (J. B. Crowe, chairman), is still studying detergency but without too much success. A summary of the results of the various series of tests made by this section will be published in the near future.

Section F on Special Detergents (C. C. Ziegler, chairman) reviewed the Standard Specifications for Trisodium Phosphate (D 538 – 39 T) and for Tetrasodium Pyrophosphate (Anhydrous) (D 595 – 40 T). The specifications for trisodium phosphate have received some criticism as having too little latitude and they will be studied further this year. It was decided to retain these specifications as tentative for another year. A basis of purchase clause together with several other minor changes is being recommended for inclusion in Specifications D 595 – 40 T as given earlier in the report.

Section G on Metal Cleaners (J. C. Harris, chairman) has developed the Proposed Methods of Chemical Analysis of Industrial Metal Cleaning Compositions which appear in an Appendix to this report. Two representative samples of metal cleaning compositions will be prepared and subjected to cooperative analysis by the members of this section to determine the efficiency of these methods. In addition to the cooperative laboratory work, it has been decided that activities on metal cleaners will be confined to the wet cleaning of aluminum. A bibliography of the test methods for the cleaning of metals will be prepared and from this it is expected that methods for the performance testing of aluminum cleaners may be devised.

Subcommittee III on Nomenclature and Definitions (C. A. Marlies, chairman):

This subcommittee has revised the definition for detergent, and presents three new definitions for inclusion in the Tentative Definitions of Terms Relating to Soaps and Other Detergents (D 459 – 41 T) as given earlier in this report.

The subcommittee also discussed the use of other soap terms such as builder, and whether they should be used with synthetic detergents. This matter as well as other definitions will be considered during the coming year.

Subcommittee IV on Papers and Publications (W. H. Koch, chairman):

After considerable discussion, this subcommittee voted to table the preparation of the monograph on alkaline detergents until a more propitious time. It was decided that there was not yet enough information available to prepare a monograph along the lines originally requested by W. G. Morse and which would do credit to the Society. The program of the subcommittee for this coming year is to prepare, with the assistance of M. L. Sheely, an article for publication in *Oil and Soap* on the analysis of commercial tetrasodium pyrophosphate, and also the analysis of tetrasodium pyrophosphate in soap. This article is to include the background of the A.S.T.M. methods and the results obtained by the cooperating laboratories prior to acceptance of the methods.

This report has been submitted to letter ballot of the committee which consists of 71 members; 45 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. P. TREVITHICK,
Chairman.

B. S. VAN ZILE,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee D-12 recommended to Committee E-10 on Standards that the Emergency Alternate Provisions in the Standard Specifications for Chip Soap (EA - D 496) and Standard Specifications for Powdered Soap (Nonalkaline Soap Powder) (EA - D 498), referred to in this report, be withdrawn. This recommendation was accepted by Committee E-10 on August 24, 1942.

APPENDIX

PROPOSED METHODS OF CHEMICAL ANALYSIS OF INDUSTRIAL METAL CLEANING COMPOSITIONS[1]

These are proposed methods and are published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia, Pa.

Scope

1. (*a*) These methods describe the procedures for the chemical analysis of industrial metal cleaning compositions in solid, paste, or liquid form. These cleaning compositions may contain caustic alkalies, silicates, phosphates, chromates, carbonates, bicarbonates, borates, sulfates, sulfites, nitrates, chlorides, soaps, rosin, sulfonated wetting agents, antifoaming agents, organic bases, organic solvents, organic coupling agents, and sometimes inorganic acid salts or organic acids. Occasionally such cleaning compositions may contain sulfonated oils; in such cases, reference should be made to the Standard Methods of Chemical Analysis of Sulfonated and Sulfated Oils (A.S.T.M. Designation: D 500) of the American Society for Testing Materials.[2] The examination under a low power microscope or magnifying glass, supplemented by a few qualitative tests, will often indicate the number of components and their identity.

(*b*) The analytical methods appear in the following order:

	Section
Preparation of Sample	2
Total Alkalinity as Na_2O	3 to 5
Total Fatty Acids (and Rosin)	6 and 7
Na_2O Combined with Fatty Acids (and Rosin)	8 and 9
Anhydrous Soap	10
Rosin (McNicoll Method):	
Qualitative	11 and 12
Quantitative	13 to 16
Total Silica Calculated as SiO_2	17 to 19
Phosphates:	
Qualitative	20 and 21
Quantitative	20 and 22
Combined Sodium and Potassium Oxides	24 and 27
Chlorides	28 to 30
Sulfates	31 to 32
Water, Distillation Method	33 to 36
Carbon Dioxide by Evolution-Absorption Method	37 to 40
Loss at 105 C	41 to 42
Total Matter Insoluble in Alcohol	43 to 45
Free Alkali	46 and 47
Synthetic Detergent or Wetting Agents	48 and 49
Volatile Hydrocarbons	50 to 53
Loss on Ignition	54 and 55
Report	56 and 57

Preparation of Sample

2. (*a*) Samples of metal cleaning compositions shall be well mixed. Excessive exposure to the air shall be avoided to prevent the sample from absorbing moisture and carbon dioxide and, if a volatile solvent is present, to prevent evaporation.

(*b*) *Solid Cleaners.*—If the cleaner is a solid and appears to contain no volatile solvent, dissolve 10.000 g. in freshly boiled distilled water and dilute to 500 ml. as the sample for analysis.

(*c*) *Liquid or Paste Cleaners.*—If the cleaner is a paste or liquid dissolve a sufficient portion in freshly boiled distilled water to give the equivalent of

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[1] These proposed methods are under the jurisdiction of the A.S.T.M. Committee D-12 on Soaps and Other Detergents. Published as information, June, 1942.

[2] 1942 Book of A.S.T.M. Standards, Part III, p. 777.

10.000 g. of the dry material in solution, and dilute to 500 ml. as the sample for analysis (Note).

NOTE.—In order to prepare this sample it is first necessary to determine the water content (Sections 33 to 36), loss at 105 C. (Sections 41 and 42), and volatile hydrocarbons (Sections 50 to 53).

TOTAL ALKALINITY AS Na_2O

Reagents

3. (*a*) *Hydrochloric Acid (0.5 N).*
(*b*) *Sodium Hydroxide (0.5 N).*
(*c*) *Ethyl Ether.*
(*d*) *Methyl Orange Indicator.*

Procedure

4. Pipette 50 ml. of the sample (1.000 g.) prepared in accordance with Section 2, into a 250-ml. Erlenmeyer flask. Add methyl orange indicator and a measured excess of 0.5 *N* HCl, and boil to expel carbon dioxide. Cool, add 25 ml. of ethyl ether, and titrate the excess HCl with 0.5 *N* NaOH. When the water layer is neutral to methyl orange, it should be clear unless sodium metasilicate (or other silicate) is present in large quantities. If silicates are present, they will form a gelatinous mass which will produce a troublesome emulsion. All the soap will have been decomposed during the reaction and the corresponding fatty acid will be found in the ether layer.

Calculation

5. Calculate the percentage of Na_2O as follows:

$$\text{Total alkalinity as } Na_2O\text{, per cent} = \frac{(A - B) \times 0.0155}{W} \times 100$$

where:

A = milliliters of 0.5 *N* HCl (1 ml. of 0.5 *N* HCl = 0.0155 g. of Na_2O),
B = milliliters of 0.5 *N* NaOH, and
W = weight of sample = 1.000 g.

TOTAL FATTY ACIDS (AND ROSIN ACIDS)

Procedure

6. Pour into a separatory funnel the neutral ethyl ether - water mixture obtained from the titration under Section 4 using additional ether to wash all the fat or oil into the separatory funnel. Allow the two layers to separate, and draw off the water layer into a second separatory funnel. Wash the water layer with two 10-ml. portions of ethyl ether. Combine the ether extracts, and wash with successive portions of distilled water until they are no longer acid to methyl orange. Transfer the ether layer to a weighed flask, evaporate the ether over a steam bath, and finally dry to constant weight at 105 C.

Calculation

7. Calculate the percentage of fatty acids (and rosin acids) as follows:

$$\text{Total fatty acids (and rosin acids), per cent} = \frac{E}{W} \times 100$$

where:

E = weight of ether residue, and
W = weight of sample = 1.000 g.

Na_2O COMBINED WITH FATTY ACIDS (AND ROSIN ACIDS)

Procedure

8. Dissolve the fatty acid (and rosin acid) residue obtained under Section 6 in warm neutral alcohol, add six drops of phenolphthalein indicator, and titrate with 0.5 *N* NaOH.

Calculation

9. Calculate the percentage of Na_2O combined with the fatty acids (and rosin acids) as follows:

$$Na_2O\text{ combined with fatty acids (and rosin acids), per cent} = \frac{B \times 0.0155}{W} \times 100$$

where:

B = milliliters of 0.5 N NaOH (1 ml. of 0.5 N NaOH = 0.0155 g. of Na_2O), and

W = weight of sample = 1.000 g.

ANHYDROUS SOAP

Calculation

10. Calculate the percentage of anhydrous soap as follows:

$$H = C \times 0.29$$
$$F = R - H$$
$$P = F + C$$

where:

C = percentage Na_2O combined with fatty acids (and rosin acids) (Section 9),

F = percentage fatty acid anhydride,

H = percentage water equivalent,

R = percentage fatty acids (and rosin acids) (Section 7), and

P = percentage anhydrous soap.

ROSIN (MCNICOLL METHOD)

Qualitative

Reagents

11. (*a*) *Acetic Anhydride, C.P.* (*99 to 100 per cent*).

(*b*) *Sulfuric Acid* (*sp. gr. 1.53*).—Prepare by adding 34.7 ml. of H_2SO_4 (sp. gr. 1.84) to 35.7 ml. of distilled water.

Procedure

12. The presence of rosin may be determined qualitatively by testing a portion of the residue obtained under Section 6 by the Liebermann-Storch reaction, as follows: Dissolve a portion of the ether extract by warming in several times its weight of acetic anhydride, and then cool the solution. Allow several drops of H_2SO_4 to flow slowly into the acetic anhydride - fat mixture. Rosin is indicated by the development of a fugitive violet coloration changing to a brownish tinge at the margin of contact of the reagents. The test should be checked by testing a sample of fatty acids to which rosin has been added. If the test for rosin is positive the quantitive procedure should be carried out as described in Sections 13 to 16.

Quantitative

Apparatus

13. The apparatus required consists of a glass flask connected, preferably by a ground-glass joint, to a reflux condenser.

(*a*) *Esterification Flask.*—A 150-ml. flask of either the round-bottom or Erlenmeyer type shall be used.

(*b*) *Reflux Condenser.*—Any suitable water-cooled, glass reflux condenser may be used.

Reagents

14. (*a*) *Potassium Hydroxide* (*0.2 N*).—Accurately standardize a 0.2 N solution of KOH in neutral redistilled alcohol (due to volatility of alcohol, this solution should be restandardized frequently).

(*b*) *Naphthalene - β-Sulfonic Acid Solution.*—Dissolve 40 g. of Eastman grade or equivalent reagent in 1 liter of c.p. absolute methyl alcohol.

(*c*) *Phenolphthalein Indicator.*—Prepare a 0.5 per cent solution in neutral redistilled alcohol.

Procedure

15. (*a*) *Preparation of Fatty and Rosin Acids.*—Dissolve a sufficient portion of the sample to give the equivalent of 50 g. of the dry material in 500 ml. of hot water. (If the cleaner to be tested contains alcohol, the alcohol should be completely removed by evaporation from the solution). Add 100 ml. of H_2SO_4 (30 per cent), heat gently until the fatty

matter collects in a clear layer. Siphon off the aqueous acid layer, add 300 ml. of hot water, boil gently for a few minutes, and siphon off the aqueous acid layer. Wash the acids in this manner three times. Complete this acidification and washing in a very short period of time, and keep the beaker covered to prevent oxidation of the acids. After the last washing, remove the last traces of water from the beaker with a pipette, filter the fatty acids through one or two thicknesses of filter paper, and dry at a temperature of 105 C. for 45 to 60 min. or heat rapidly to 130 C. and allow to cool. Do not hold at 130 C., but if water is present, decant the clear fatty acids into another beaker, and again reheat them momentarily to 130 C. These acids may then be used for the rosin determination.

(*b*) *Esterification and Titration.*—Weigh about 2 ± 0.001 g. of the fatty acids into the esterification flask. Add 25 ml. of naphthalene - β-sulfonic acid solution. Add a few glass beads to ensure smooth boiling, attach the reflux condenser, and boil for 30 min.; also, run a blank test using 25 ml. of the reagent. At the end of the boiling period cool the contents of the flask, add 0.5 ml. of phenolphthalein indicator, and titrate immediately with 0.2 N alcoholic potassium hydroxide.

Calculation

16. (*a*) Calculate the results as follows:

$$R = \frac{(S - B) \times N \times 0.346 \times 100}{W}$$

$$R_1 = R - 1.0$$

$$R_2 = \frac{R_1 \times F}{100}$$

$$R_S = \frac{R_1 \times 1.064 \times A}{100}$$

where:

R = percentage of rosin acids in fatty acids,

R_1 = corrected percentage of rosin acids in fatty acids (Note),

R_2 = percentage of rosin on basis of original sample,

R_S = percentage of rosin soda soap on basis of original sample,

S = milliliters of KOH required to titrate sample,

B = milliliters of KOH required to titrate blank,

N = normality of KOH,

W = weight of sample in grams,

F = percentage of total fatty acids (and rosin acids) in the cleaner, and

A = percentage of total anhydrous soap (Section 10).

(*b*) If true fatty acid soap is desired, subtract the rosin soap from the total anhydrous soap.

NOTE.—Coöperative studies have shown that the McNicoll method gives results approximately 1 per cent higher than the amount of rosin present. Consequently, the committee recommends deducting 1 per cent from the percentage of rosin found in the fatty acids.

TOTAL SILICA CALCULATED AS SiO_2

Preparation of Sample

17. (*a*) *When Soap is Present.*—If soap is present in the cleaner, draw off the water solution from the total alkalinity titration (Section 4) into a porcelain evaporating dish. Add an excess of HCl (sp. gr. 1.19), evaporate the mixture to dryness on a steam bath, and carry out the procedure as described in Section 18. The weight of the sample will be 1.000 g.

(*b*) *When Soap is Not Present.*—If soap is *not* present in the cleaner, transfer a 100-ml. aliquot of the sample solution obtained as described in Section 2, to a 400-ml. evaporating dish, neutralize with HCl (sp. gr. 1.19) using methyl orange as the indicator, evaporate to apparent dryness on a steam bath, and carry out the procedure as described in Section 18. The weight of the sample will be 2.000 g.

Procedure

18. (*a*) Triturate the dehydrated residue, obtained as described in Section 17 (*a*) or (*b*), with the smooth end of a stirring rod, moisten the residue with 10 ml. of diluted HCl (1:1), and again evaporate to apparent dryness on the steam bath. Dehydrate at 110 C. for 1 hr., take up the residue with 10 ml. of diluted HCl (1:1) and 20 ml. of distilled water, and digest a short time on the steam bath to effect solution of the soluble salts. Filter the silica on a No. 40 Whatman filter paper or equivalent, by washing the dish with hot distilled water. Scrub the dish with a rubber policeman and again wash thoroughly with hot distilled water. Wash the residue and paper free from acid with hot distilled water and reserve.

(*b*) Evaporate the filtrate and washings on the steam bath in the porcelain dish used before, moisten the residue with 10 ml. of diluted HCl (1:1), and again evaporate to dryness. Dehydrate at 110 C. for 1 hr., take up the residue with 10 ml. of diluted HCl (1:1) and 20 ml. of distilled water, digest as before to dissolve soluble salts, and filter off any additional silica on a separate filter paper. Scrub the dish and wash the residue and filter paper free from acid as before. Reserve the filtrate for the determination of phosphates (Section 22) and sulfates (Section 31). Transfer both papers and residues to a platinum crucible previously ignited and weighed without cover and ignite until free from carbon, heating slowly at first. Cover the crucible with a platinum cover, heat to the highest temperature of a blast lamp for 15 min., cool in a desiccator, and weigh without the crucible cover, weight *A*. Moisten the weighed contents of the crucible with water, add 10 ml. of HF and 4 drops of strong H_2SO_4, evaporate to dryness over a low flame, ignite at the highest temperature of the blast lamp for 2 min., cool in a desiccator, and weigh, weight *B*.

Calculation

19. Calculate the weight of the total silica as SiO_2 as follows:

$$\text{Total SiO}_2\text{, per cent} = \frac{\text{wt. } A - \text{wt. } B}{\text{wt. of sample}} \times 100$$

PHOSPHATES

Reagents

20. (*a*) *Nitric Acid (0.5 N).*

(*b*) *Sodium Hydroxide (50 per cent by Weight).*—When using, decant the solution from the settled carbonate.

(*c*) *Sodium Hydroxide (0.5 N).*

(*d*) *Silver Nitrate (0.85 N).*

(*e*) *Ammonium Hydroxide Solution (sp. gr. 0.96 or Approximately 6 N.)*—Mix 400 ml. of NH_4OH (sp. gr. 0.90) with 600 ml. of distilled water. Check with a hydrometer or by titration.

(*f*) *Molybdate Solution.*—Mix 100 g. of pure molybdic anhydride or 118 g. of molybdic acid (85 per cent) with 400 ml. of distilled water, and add 80 ml. of NH_4OH (sp. gr. 0.90). When solution is complete, filter, and pour the solution slowly, while constantly stirring into a mixture of 400 ml. of HNO_3 (sp. gr. 1.42) and 600 ml. of distilled water. Allow the solution to settle for 24 hr., and use the clear supernatant liquid.

(*g*) *Potassium Nitrate Solution.*—Dissolve 10 g. of KNO_3 in freshly boiled distilled water, dilute to 1000 ml. with freshly boiled distilled water, and mix.

Qualitative

Procedure

21. The presence of an orthophosphate (Na_3PO_4) or pyrophosphate ($Na_4P_2O_7$) may be confirmed as follows: Acidify a sample of the cleaner with diluted HNO_3 (1:5). Extract fatty acids, if formed, with ether, remove carbon dioxide by aeration of the solution, and neutralize with clarified NaOH (50 per cent) until faintly pink to phenolphthalein. Add silver nitrate (0.5 *N*) to the solution. The appearance of either the yellow silver orthophosphate precipitate, or the white silver pyrophosphate precipitate indicates the presence of phosphates. Precipitates may also be obtained with chlorides and chromates. If phosphates are present, they shall be determined in accordance with the quantitative procedure described in Section 22.

Quantitative

Procedure

22. To the water solution obtained from the determination of total silica contained in a 250-ml. flask or larger (Section 18 (*b*)), add 5 to 10 ml. of HNO_3 (sp. gr. 1.42), and evaporate to dryness on a steam bath. Dissolve the residue in diluted HNO_3 (1:5), and boil. This will convert any pyrophosphates or polyphosphates to the ortho state. To the boiled and cooled solution, or an aliquot if necessary, containing not more than 0.08 g. of P_2O_5, add a small piece of litmus paper, and carefully neutralize with NH_4OH (sp. gr. 0.96), dilute to 75 ml., and then add 25 ml. of HNO_3 (sp. gr. 1.42). Add to this solution 40 ml. of NH_4OH (sp. gr. 0.96) and 60 ml. of molybdate solution, stopper, shake for 10 min., and allow to stand at room temperature until the precipitate settles. Filter through an 11-cm. filter paper of close texture, wash the flask and precipitate five times with 15 to 20-ml. portions of potassium nitrate solution (1 per cent), then wash the filter paper containing most of the precipitate five times with similar portions of the same solution. Wash the paper carefully each time from the rim downward and then allow to drain completely before washing with the next portion of solution. The wash solution, as well as others subsequently used, shall be free of CO_2. Return the paper and precipitate to the flask, add enough 0.5 *N* NaOH until the yellow precipitate disappears, and then approximately 1 ml. in excess. Dilute with 25 ml. of distilled water, stopper, and shake until the precipitate is dissolved. Wash off the stopper with distilled water, dilute the solution to about 150 ml. with distilled water, add 6 drops of phenolphthalein solution (1 per cent), and discharge the pink color with 0.5 *N* HNO_3. Finish the titration by adding 0.5 *N* NaOH until the reappearance of the pink color.

Calculation

23. Calculate the percentage of phosphates as follows:

$$P_2O_5\text{, per cent} = \frac{\text{ml. of 0.5 } N \text{ NaOH} \times 0.001542}{\text{wt. of sample}} \times 100$$

$$Na_3PO_4\text{, per cent} = P_2O_5\text{, per cent} \times 2.3101$$

$$Na_4P_2O_7\text{, per cent} = P_2O_5\text{, per cent} \times 1.8734$$

COMBINED SODIUM AND POTASSIUM OXIDES

Reagents

24. (*a*) *Ammonium Chloride Solution.*—Dissolve 100 g. of NH_4Cl in 500 ml. of distilled water, add 5 to 10 g. of pulverized K_2PtCl_6, and shake at intervals

for 6 to 8 hr. Allow the mixture to settle overnight, and filter. (The residue may be used for the preparation of a fresh supply of NH_4Cl solution.)

(*b*) *Platinum Solution.*—Prepare a solution containing the equivalent of 1 g. of metallic platinum (2.1 g. of H_2PtCl_6) in each 10 ml. of solution. For materials containing less than 15 per cent of K_2O, a solution containing 0.2 g. of metallic platinum (0.42 g. of H_2PtCl_6) in each 10 ml. of solution is recommended.

Preparation of Sample

25. Weigh a sufficient portion of the sample to give the equivalent of 10 ± 0.01 g. of the dry material and sinter it in an evaporating dish below a dull red heat. Leach the ash with hot water, filter into a 100-ml. volumetric flask, and wash the paper with three 5 to 10-ml. portions of hot water. Complete the ashing after returning the filter paper and residue to the original dish and sintering as before. Excessive heating shall be avoided. Removal of most of the alkali present by thoroughly washing the ash with hot water before completion of the ashing will aid in preventing overheating of the greater portion of the sample. Add a few drops of HCl (1:1) to the ash and wash the contents of the dish into the volumetric flask. Acidify the solution in the volumetric flask with HCl, dilute to 100 ml., mix thoroughly, and pass through a dry filter.

Procedure

26. Acidify an accurately measured 10-ml. aliquot of the solution obtained in Section 25 with a few drops of HCl and add 10 ml. of the platinum solution. Evaporate the solution on a water bath to a thick paste which will become solid on cooling to room temperature. Avoid exposure to ammonia fumes while heating the solution. Treat the residue with approximately 6 ml. of 80 per cent ethyl alcohol and add 0.6 ml. of HCl (sp. gr. 1.19). Filter on a Gooch crucible and wash the precipitate thoroughly with 80 per cent ethyl alcohol both by decantation and on the filter, continuing the washing until after the filtrate is colorless. Then wash the residue five or six times with 25-ml. portions of the NH_4Cl solution to remove the impurities from the precipitate. Wash again thoroughly with 80 per cent ethyl alcohol, dry the precipitate at 100 C. for 30 min., and weigh.

Calculation

27. (*a*) Calculate the percentage of potassium oxide as follows:

$$K_2O\text{, per cent} = \frac{\text{wt. of } K_2PtCl_6 \times 0.19376}{10\text{ g.}} \times 100$$

(*b*) Calculate the percentage of sodium oxide as follows:

$$Na_2O\text{, per cent} = C - (D \times 0.6582)$$

where:

C = percentage total alkalinity as Na_2O (Section 5), and

D = percentage combined K_2O (Paragraph (*a*)) converted to equivalent Na_2O.

CHLORIDES

Reagents

28. (*a*) *Silver Nitrate Solution (0.1 N).*

(*b*) *Magnesium Nitrate Solution (20 per cent).*—Dissolve 200 g. of chloride-free $Mg(NO_3)_2 \cdot 6H_2O$ in 1 liter of water.

(*c*) *Potassium Chromate Indicator.*—Dissolve 5 g. of K_2CrO_4 in water and add solution of silver nitrate until a slight red precipitate is produced, filter the solution, and dilute to 100 ml.

Procedure

29. Dissolve a sufficient portion of the sample to give the equivalent of 5 ± 0.01 g. of the dry material in 300 ml. of water, boiling if necessary to effect solution. Add an excess of neutral, chloride-free magnesium nitrate solution (about 25 ml. of the 20 per cent $Mg(NO_3)_2 \cdot 6H_2O$ solution). Without cooling or filtering, titrate with standard $AgNO_3$, using 2 ml. of K_2CrO_4 indicator.

Calculation

30. Calculate the chlorides as percentage of sodium chloride as follows:

$$\text{NaCl, per cent} = \frac{\text{ml. of } 0.1\ N\ AgNO_3 \times 0.00585}{\text{wt. of sample}} \times 100$$

SULFATES

Procedure

31. (*a*) The matter insoluble in alcohol obtained in accordance with Section 44 may suffice, or the filtrate from the determination of total silica as SiO_2 (Section 18 (*b*)) may be used as the sample.

(*b*) If only a determination for sulfates is desired, ignite 10 ± 0.10 g. of the sample and use the ash from the ignition. Digest with 100 ml. of water, cover with a watch glass, and carefully neutralize to methyl orange with HCl. When neutralized, add 5 ml. excess of HCl, filter, and wash the residue thoroughly. (Evaporation to dryness is unnecessary unless gelatinous silica should have separated. Evaporation should never be performed on a bath heated by gas.)

(*c*) Dilute the filtrate to 250 ml. in a beaker, and boil. To the boiling solution add 15 to 20 ml. of $BaCl_2$ (10 per cent) slowly drop by drop from a pipette. Continue boiling until the precipitate is well formed, or digest on a steam bath overnight. Set aside overnight or for a few hours, filter through a prepared Gooch crucible, ignite gently, and weigh as $BaSO_4$.

Calculation

32. Calculate the sulfates as percentage of sodium sulfate as follows:

$$Na_2SO_4\text{, per cent} = \frac{\text{wt. of } BaSO_4 \times 0.60858}{\text{wt. of sample}} \times 100$$

WATER, DISTILLATION METHOD

Apparatus

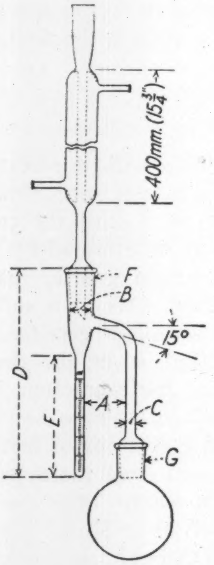
33. The apparatus required consists of a glass flask heated by suitable means and provided with a reflux condenser discharging into a trap and connected to the flask. The connections between the trap and the condenser and flask shall be interchangeable ground joints. The trap serves to collect and measure the condensed water and to return the solvent to the flask. A suitable assembly of the apparatus is illustrated in Fig. 1.

(*a*) *Flask.*—A 500-ml. flask of either the short-neck, round-bottom type or the Erlenmeyer type shall be used.

(*b*) *Heat Source.*—The source of heat may be either an oil bath (stearic acid, paraffin wax, etc.), or an electric heater provided with a sliding rheostat or other means of heat control.

(*c*) *Condenser.*—A water-cooled glass reflux condenser (Fig. 1), having a jacket approximately 400 mm. ($15\frac{3}{4}$ in.) in length with an inner tube 9.5 to 12.7 mm. ($\frac{3}{8}$ to $\frac{1}{2}$ in.) in outside diameter shall be used. The end of the condenser to be inserted in the trap may be ground off at an angle of 30 deg. from the vertical axis of the condenser. When inserted into the trap, the tip of the condenser shall be about 7 mm. ($\frac{1}{4}$ in.) above the surface of the liquid in the trap after the distillation conditions have been established. Figure 1 shows a conventional sealed-in type of condenser, but any other condenser fulfilling the detailed requirements above may be used.

(*d*) *Trap*.—A trap made of well-annealed glass constructed in accordance with Fig. 1 and graduated as shown to contain 5 ml. at 20 C. shall be used. It shall be subdivided into 0.1-ml. divisions, with each 1-ml. line numbered (5 ml. at top). The error in any indicated capacity may not be greater than 0.05 ml.



A—45 to 55 mm.
B—22 to 24 mm., inside diameter
C—9 to 11 mm., inside diameter
D—235 to 240 mm.
E—146 to 156 mm.
F and G are interchangeable joints, standard taper 24/40.

FIG. 1.—Assembly of Distillation Apparatus.

Reagent

34. *Xylene (or Toluene)*.—(For cleaners containing 1 per cent or more of glycerol, toluene shall be used instead of xylene.) Saturate the xylene (or toluene) with water by shaking with a small quantity of water and distill. Use the distillate for the determination.

Procedure

35. For cleaners containing from 5 to 25 per cent moisture and volatile matter use 20 ± 0.04 g. of the sample. For cleaners containing more than 25 per cent moisture and volatile matter use 10 ± 0.02 g. of the sample. Carefully transfer the weighed sample to the 500-ml. flask. Add approximately 10 g. of anhydrous, fused sodium acetate to prevent violent frothing, and then follow with 100 ml. of xylene (or toluene) which has been saturated with water. Attach the flask to the trap which is connected to the condenser. Prior to starting the determination, fill the receiver with saturated xylene (or toluene) by pouring in through the reflux condenser. So that the refluxing will be under better control, wrap the flask and tube leading to the receiver with asbestos cloth. Heat the oil bath with a gas burner or other source of heat, or apply heat directly to the flask with an electric heater and distill slowly. The rate at the start should be approximately 100 drops per minute. When the greater part of the water has distilled over, increase the distillation rate to 200 drops per minute until no more water is collected. Purge the reflux condenser during the distillation with 5-ml. portions of xylene (or toluene) to wash down any moisture adhering to the walls of the condenser. The water in the receiver may be made to separate from the xylene (or toluene) by using a spiral copper wire. Move the wire up and down in the condenser occasionally, thus causing the water to settle at the bottom of the receiver. Reflux for at least 2 hr., and shut off the heat at the end of this period. Adjust the temperature of the distillate to 20 C., and read the volume of water.

Calculation

36. Calculate the percentage of water as follows:

$$\text{Water, per cent} = \frac{V \times 0.998}{W} \times 100$$

where:

V = volume of water in milliliters at 20 C., and

W = weight of the sample in grams.

CARBON DIOXIDE BY EVOLUTION-ABSORPTION METHOD

Apparatus

37. The apparatus shall consist of the following:

(*a*) *Extraction Flask.*—A 150-ml. wide-neck extraction flask.

(*b*) *Condenser.*—A 25-cm. reflux condenser.

(*c*) *Thistle Tube.*—A thistle tube equipped with a two-way stopcock, the end to be drawn out to a fine point.

(*d*) *U-Tubes.*—Four U-tubes prepared as described in Section 38.

(*e*) *Drying Tube.*—A drying tube filled with a drying agent as described in Section 38 (*b*).

(*f*) *Aspirator.*—A means of aspirating the apparatus to ensure a continuous stream of CO_2-free air.

Preparation of Apparatus

38. (*a*) *Apparatus Assembly.*—Place the 150-ml. extraction flask on a gauze over a burner. Fit the flask with a three-hole rubber stopper, one opening to carry the reflux condenser, the second to carry a thistle tube with a two-way stopcock for the introduction of acid into the flask, and the third to carry the tube for the introduction of a continuous stream of CO_2-free air into the flask. Draw out the ends of the thistle and air supply tubes to a small point, and place them in the stopper so that the points are very close to the bottom of the flask. Attach to the air supply tube, one of the U-tubes containing ascarite so that the air admitted to the flask will be free from CO_2.

(*b*) *Preparation of Absorption Train.* Attach to the top of the reflux condenser a train consisting of the following: (*1*) a U-tube containing granulated zinc for the removal of acid gases, (*2*) a drying tube containing magnesium perchlorate, drierite, or anhydrous calcium chloride, (*3*) a weighed U-tube containing ascarite in the first half and the same drying agent in the second half as used in (*2*), and (*4*) a protective U-tube containing any of the above mentioned drying agents. Attach the final tube to an aspirator.

Procedure

39. Aspirate with a stream of CO_2-free air at a rate of approximately 20 to 30 ml. per min. until the train is free from CO_2 as determined by no further change in weight greater than 0.3 mg. in the U-tube. Weigh a sufficient portion of the sample to give the equivalent of 10 ± 0.01 g. of the dry material directly into the extraction flask, cover with 50 ml. of freshly boiled distilled water, add two drops of methyl orange indicator, and close the apparatus with the train in place. Start the aspiration at a rate of 20 to 30 ml. per min., and slowly add through the thistle tube sufficient 6 N H_2SO_4 to neutralize the NaOH and a sufficient excess to ensure the final acidity of the mixture as indicated by the methyl orange. Always leave some acid in the thistle tube as an air seal. Heat gently and continue until the contents of the flask have boiled for 5 min.; remove the source of heat, and continue aspirating until the flask has cooled, or for about 30 min. Remove the U-tube containing ascarite and weigh using a tared U-tube as a counterpoise. The increase in weight represents CO_2.

Calculation

40. (*a*) From the increase in weight

of the tube calculate the percentage of Na_2CO_3 as follows:

$$Na_2CO_3\text{, per cent} = \frac{\text{wt. of } CO_2 \times 2.409}{\text{wt. of sample}} \times 100$$

(*b*) Calculate the percentage of CO_2 as follows:

$$CO_2\text{, per cent} = \frac{\text{wt. of } CO_2}{\text{wt. of sample}} \times 100$$

LOSS AT 105 C.

Procedure

41. Dry 2 to 10 ± 0.01 g. of the sample at 105 C. to constant weight, care being taken to prevent the sample from absorbing CO_2. Reserve the residue for the determination of total matter insoluble in alcohol (Section 44).

Calculation

42. Calculate the percentage loss at 105 C. as follows:

$$\text{Loss at 105 C., per cent} = \frac{\text{wt. of dried sample}}{\text{wt. of sample}} \times 100$$

NOTE.—Bicarbonates are converted to carbonates on drying at 105 C. and lose 36.9 per cent of their weight. It is next to impossible to determine bicarbonates in the presence of caustic alkalies because of conversion to the carbonate.

TOTAL MATTER INSOLUBLE IN ALCOHOL

Reagent

43. *Ethyl Alcohol* (*95 per cent*).—Freshly boiled ethyl alcohol, 95 per cent or higher and neutral to phenolphthalein, either Formula No. 3-A or No. 30 of the U. S. Internal Revenue Bureau.

Procedure

44. Digest the residue, obtained in accordance with Section 41, with 200 ml. of freshly boiled, neutral alcohol in a covered vessel on a steam bath until soap or wetting agent is dissolved. Filter through a counterpoised filter paper, neutral to phenolphthalein, or through a weighed Gooch crucible with suction, protecting the solution from carbon dioxide and other acid fumes during the operation by covering with a watch glass. If a filter paper is used weigh in a counter balanced ointment box or like container to prevent moisture absorption while weighing. Wash the residue on the paper or in the crucible with hot neutral alcohol until free from soap, and reserve the filtrate and washings. Dry the filter paper or crucible with the residue at 105 C. for 3 hr., cool, and weigh the total matter insoluble in alcohol. Reserve the alcoholic filtrate for determination of free alkali (Section 46).

Calculation

45. Calculate the percentage of total matter insoluble in alcohol as follows:

$$T = \frac{M}{W} \times 100$$

where:

T = percentage of total matter insoluble in alcohol,
M = weight of insoluble matter, and
W = weight of sample in grams.

FREE ALKALI

Procedure

46. Heat the reserved filtrate from the determination of total matter insoluble in alcohol (Section 44) to incipient boiling, add 0.5 ml. of a 1 per cent solution of phenolphthalein, titrate with 0.5 N HCl. Reserve the titrated solution for the determination of the synthetic detergent or wetting agent (Section 48).

Calculation

47. Calculate the percentage of free alkali as NaOH as follows:

$$\text{Free alkali as NaOH, per cent} = \frac{\text{ml. of } 0.5\ N\ \text{HCl} \times 0.0200}{\text{wt. of sample}} \times 100$$

SYNTHETIC DETERGENT OR WETTING AGENT

Procedure

48. Transfer the titrated solution obtained in accordance with Section 46 to a tared beaker or evaporating dish, and evaporate to dryness on a steam bath. Dry to constant weight at 105 C.

Calculation

49. Calculate the percentage of synthetic detergent or wetting agent as follows (Note):

$$A = \frac{E - (G + H)}{W} \times 100$$

where:

A = percentage synthetic detergent or wetting agent,
E = weight of titrated alcohol extract,
G = weight of NaCl,
H = weight of soap and rosin soap, and
W = weight of sample in grams.

NOTE.—Since soap and rosin soap are also alcohol soluble, and since the known amount of NaCl was formed by the titration in the determination of free alkali (Section 46), also determined in the procedure for chlorides (Section 29), correction for soap, rosin soap, and chlorides may be made for each of these constituents. One milliliter of 0.5 N HCl (see Section 46) is equivalent to 0.0292 g. of NaCl.

VOLATILE HYDROCARBONS

50. This procedure requires a source of dry, oil-free steam which is passed through the sample treated with acid, sufficient to liberate the fatty acids from the cleaner. The steam is next passed through strong NaOH solution to scrub out any volatile fatty acids, while the volatile hydrocarbons are condensed with the steam in a suitable arrangement which allows the excess water to flow away, leaving the volatile hydrocarbons in the measuring burette. The procedure may be applied to samples containing substances immiscible with water and volatile with steam. (For solvents heavier than water the trap as shown in Fig. 1 for determining water by the distillation method (Sections 33 to 36) should be used.)

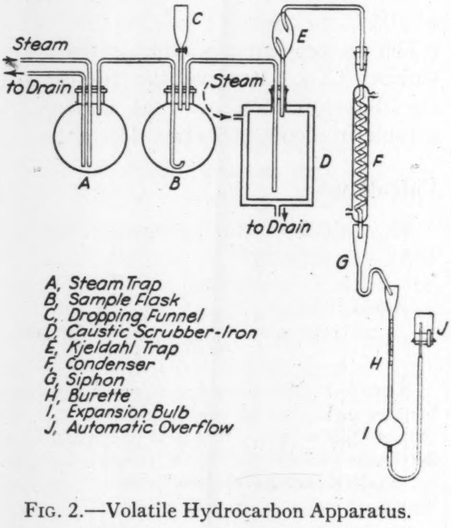


FIG. 2.—Volatile Hydrocarbon Apparatus.

Apparatus

51. The apparatus and its arrangement are shown in Fig. 2. The following are the important items:

(a) *Steam Trap, A.*—A 1-liter round-bottom, ring-neck flask equipped with a siphon tube to the drain from the bottom of the flask and provided with a means of regulating the steam flow into the flask.

(b) *Evolution or Sample Flask, B.*—A 1-liter round-bottom ring-neck flask. In case large samples are desirable the size of this flask may be increased.

(*c*) *Caustic Scrubber Flask, D.*—A steam jacketed metal flask is preferred, but a 1-liter Florence flask provided with a steam coil of 0.32 cm. (0.125 in.) copper tubing around the upper half may be used. If the glass flask is used it should be provided with a safety bucket below it and should be renewed frequently since the strong caustic dissolves the glass rather rapidly. This flask should be connected to the condenser by a Kjeldahl connecting tube, *E*, or similar safety device.

The inlet for the steam into the evolution and scrubber flasks should extend nearly to the bottom of the flasks and should be bent at right angles and parallel to the sides of the flask.

(*d*) *Condenser, F.*—A 30.5-cm. (12-in.) or longer spiral condenser of sufficient bore so the condensate will not readily close it.

(*e*) *Measuring Burette, H.*—A 10-ml. burette calibrated to 0.1 ml. and carrying a bulb, *I*, of approximately 100-ml. capacity, at the lower end. If desired, an ordinary 10-ml. Mohr type burette may be used having attached to it by rubber tubing a bulb of proper capacity which has been blown in the laboratory. An ordinary burette funnel may be placed in the top of the burette in place of the special flared-out top shown in Fig. 2.

The stoppers used should be of a good grade of rubber and should have been thoroughly cleaned free from any surface sulfur and should be given a steam distillation in position for several hours before use on a sample.

Insulating the flasks and tubing to reduce condensation aids distillation and its control.

Procedure

52. (*a*) Place 150 ml. of NaOH solution (approx. sp. gr. 1.47) and several sticks of solid NaOH to provide against dilution in the scrubber flask. Rinse out the condenser and burette with acetone. Attach a rubber tubing to the lower end of the burette, fill the burette and tubing with water, and raise the outer end of the tubing so that the water level in the burette is near the top of the scale when the water is flowing to the drain from the automatic overflow, *J*. Be sure that the connections are tight and that the tubing contains no air bubbles. Place the condenser in position so that the lower end extends directly into the upper end of the burette just above the water level or connect to an adapter siphon, *G*, which discharges into the burette. The cooling water should be 15.5 C. or colder. Ice water may be desirable for low-boiling hydrocarbons.

(*b*) Weigh a sufficient portion of the sample to give the equivalent of 50 g. of the dry material, and transfer to the evolution flask (Note). Add about 10 g. of gum arabic (commercial) and 100 ml. of distilled water. Place the flask in position with 100 ml. of H_2SO_4 (1:3) in a dropping funnel, *C*, carried in the stopper. Connect with the steam and wash the flasks and the condenser, making sure that the stoppers are tightly fitting and held in place by wiring. Unnecessary rubber connections in the lines between the evolution flask and condenser should be avoided by placing the glass portions as tightly together as possible.

NOTE.—For some samples the volatile hydrocarbon content may be so low that a larger sample than 50 or 100 g. is desirable. The size of the evolution flask may need to be increased if larger samples are used. The amount of water in the evolution flask and acid used should also be correspondingly increased.

(*c*) Add the acid to the sample slowly to avoid excessive frothing. While adding the acid, turn on the steam cautiously, so adjusting the pressure by a bleeder valve that just enough steam flows to prevent any liquid from backing into the steam trap flask.

(*d*) When all the acid has been added, turn on enough steam to cause brisk distillation, taking care that no liquid is carried over from the evolution and wash flasks and that the condenser water does not become warm.

(*e*) Continue the distillation until there is no increase in the volume of the upper layer for 45 min. or no small droplets can be noted in the condensate.

(*f*) When distillation is completed, shut off and drain the condenser water, and allow the steam to heat up the condenser to drive out the last traces of volatile hydrocarbon. Shut off the steam as soon as vapor begins to issue from the lower end of the condenser. Immediately open the stopcock of the dropping funnel to prevent NaOH being drawn into the evolution flask.

(*g*) Stopper the burette and allow its contents to come to room temperature or bring them to a definite temperature by immersing the burette for 1 to 2 hr. in a water bath held at 25 C.

(*h*) Read the volume of the upper layer to the nearest 0.01 ml. The volume multiplied by the specific gravity equals the weight of the volatile hydrocarbon. The specific gravity should be determined at the temperature at which the volume is read. A small Sprengel tube made of 3-mm. glass tubing is convenient for this purpose.

Calculation

53. Calculate the percentage of volatile hydrocarbons as follows:

$$V = \frac{M \times \text{sp. gr.}}{W} \times 100$$

where:

V = percentage of volatile hydrocarbons,
M = milliliters of volatile hydrocarbon, and
W = weight of sample in grams.

LOSS ON IGNITION

Procedure

54. Weigh accurately about 2 g. of sand in a clean platinum crucible with a tight-fitting lid, and ignite to constant weight. Weigh accurately about 2 g. of the cleaner in the crucible, and heat with a low flame. Increase the heat gradually as the water is driven off, care being taken to prevent spattering. Ignite to constant weight. Cool in a desiccator, and weigh.

Calculation

55. Calculate the loss on ignition as follows:

$$\text{Loss on ignition, per cent} = \frac{\text{loss in wt.}}{\text{wt. of sample}} \times 100$$

REPORT

Composition of Cleaner

56. (*a*) *Paste or Liquid Cleaners.*—The sum of the water, soap (and rosin soap), volatile hydrocarbons, synthetic detergent or wetting agent, and total matter insoluble in alcohol shall be reported and should represent 100 per cent but seldom totals this amount. The constituents of the total matter insoluble in alcohol may be reported as described in Paragraph (*b*) for the constituents of solid cleaners

(*b*) *Solid Cleaners.*—The following constituents shall be reported:

(*1*) Percentage of total Na_2O,

(*2*) Percentage of K_2O,

(*3*) Percentage of free NaOH,

(*4*) Percentage of water (distillation method), (*5*) Percentage of loss at 105 C.,

(*6*) Percentage of loss on ignition,

(*7*) Percentage of matter insoluble in alcohol,

(*8*) Percentage of fatty acids (and rosin acids),

(*9*) Percentage of rosin,

(*10*) Percentage of synthetic detergent or wetting agent,

(*11*) Percentage of SiO_2,

(*12*) Percentage of P_2O_5,

(*13*) Percentage of CO_2,

(*14*) Percentage of NaCl,

(*15*) Percentage of Na_2SO_4, and

(*16*) Percentage of volatile hydrocarbons.

NOTE.—Of the above determinations water, free NaOH, loss at 105 C., loss on ignition, matter insoluble in alcohol, rosin, synthetic detergent or wetting agent, NaCl, Na_2SO_4, and volatile hydrocarbons are complete determinations in themselves. Water may be free or combined as water of crystallization. Loss at 105 C. will show the total free or uncombined volatile matter, while loss on ignition will indicate the amount of combined water and organic or inorganic matter present. The determination of material insoluble in alcohol will account for most of the alkalies except sodium hydroxide, and is a means for checking the matter other than soap or synthetic agent which may be present in a composition. Since rosin is often added as such to metal cleaning compositions, it is not always satisfactory to calculate it as rosin soap.

Conversion Factors

57. Conversion of the various constituents determined to their equivalents as chemical compounds for the purpose of reporting may be made as follows:

(*a*) *Soap.*—See Section 10.

(*b*) *Sodium Silicate.*—To calculate Na_2O combined with SiO_2:

Use ratio of 1 Na_2O to 3.25 SiO_2 for liquids and pastes. This silicate is most commonly used:

Na_2O, per cent = SiO_2, per cent × 0.3077 . (1)

SiO_2 to Na_2SiO_3, if identified:

Na_2O, per cent = 0.5082 × Na_2SiO_3, per cent . (2)

SiO_2 to $(Na_2O)_{1.5}$ (SiO_2), if identified:

Na_2O, per cent
= 0.6075 × sesquisilicate per cent . . (3)

SiO_2 to Na_4SiO_4, if identified:

Na_2O, per cent
= 0.6737 × Na_4SiO_4, per cent . . (4)

(*c*) *Sodium Phosphate.*—To calculate Na_2O combined with P_2O_5:

Calculate P_2O_5 to Na_3PO_4, if identified:

Titratable Na_2O, per cent
= 0.3778 × Na_3PO_4, per cent . . (5)

Calculate P_2O_5 to $Na_4P_2O_7$, if identified:

Titratable Na_2O, per cent
= 0.2331 × $Na_4P_2O_7$, per cent . . (6)

Calculate P_2O_5 to Na_3PO_4 and Na_4P_2,O_7 if identified.

NOTE.—If the total P_2O_5 is known, and the sum of the two phosphates, (this can often be obtained by subtracting the total of all the other known compounds from 100, but may be far from accurate because of uncertainties surrounding such items as carbonates, silicates, etc.) this is a simple algebraic calculation.

Calculate percentage of Na_2O as in Eqs. (5) and (6)

(*d*) *Potassium.*—If potassium was found, it is very likely to be present as potassium soap. Calculate the K_2O, if present, to soap in about the correct amount to combine with fatty acids:

Mol. wt. of K soap = $(F + 56.1) - 18$ . . (7)

where F = molecular weight of fatty acids (and rosin acids) (Section 7)

Wt. of K soap =

$$\frac{2 \times \text{mol. wt. of K soap} \times K_2O\text{, per cent}}{94.2} \quad . . (8)$$

K soap, per cent =

$$\frac{\text{wt. of K soap}}{\text{wt. of sample (1.000 g.)}} \times 100 \quad . . (9)$$

(*e*) *Residual K_2O.*—Calculate residual K_2O, if any remains to K_2CO_3:

K_2CO_3, per cent = K_2O, per cent × 1.467 . . . (10)

(*f*) *Sodium and Potassium Oxides.*—Calculate CO_2 to Na_2CO_3 if no K_2CO_3 is present:

$$Na_2CO_3\text{, per cent} = CO_2\text{, per cent} \times 2.409 \quad (11)$$

If K_2CO_3 is present calculate the percentage of CO_2 combined with K_2O as follows:

$$CO_2\text{, per cent} = K_2CO_3\text{, per cent} \times 0.3184 \quad (12)$$

Then subtract this percentage of CO_2 from the total CO_2 and calculate the balance to Na_2CO_3 as in Eq. 10.

(*g*) *Total Na_2O.*—Total the calculated percentage of Na_2O combined with all the alkali titratable acid radicals:

		Na_2O, per cent
Soap (Section 10)		———
Sodium silicate, (Paragraph (*b*))	1:3.25 ratio (Eq. 1) or	———
	1:1 ratio, if identified (Eq. 2), or	———
	1.5:1 ratio, if identified (Eq. 3), or	———
	2:1 ratio, if identified (Eq. 4)	———
Sodium phosphate[a] (Paragraph (*c*))	Na_3PO_4 (Eq. 5)	———
	$Na_4P_2O_7$ (Eq. 6)	———
K_2CO_3, (Paragraph (*d*))	K_2CO_3, per cent × 0.4485 (equivalent to Na_2O) (Eq. 10)	———
Na_2CO_3, (Paragraph (*e*))	Na_2CO_3, per cent × 0.5848 (Eq. 11)	———
Total		———

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[a] One Na for each P is not titratable.

(*h*) If this total is in excess of the total alkalinity as percentage of Na_2O determined in accordance with Sections 4 and 5, the wrong silicate or phosphate has been used in the calculations.

(*i*) If the total calculated percentage of Na_2O is less than the total Na_2O as determined under Sections 4 and 5 and sodium hydroxide has been proven to be present, calculate the residual alkalinity to NaOH as follows:

$$NaOH\text{, per cent} = Na_2O\text{, per cent} \times 1.2903$$

REPORT OF COMMITTEE D-13

ON

TEXTILE MATERIALS

Committee D-13 on Textile Materials held two meetings during the year: one on October 15 to 17, 1941, in New York, N. Y., and the other on March 11 to 13, 1942, in Providence, R. I. The number of members and guests registered was 177 and 139, respectively. The total attendance at these two meetings set a new high record. Of the 28 subcommittees or sections which compose the organization of Committee D-13, 16 held sessions at the fall meeting and 17 at the spring meeting. The executive direction of the committee's affairs required two meetings of the officers and two meetings of the Advisory Committee.

The above facts, together with the numerous subcommittee activities and the many recommendations regarding standards reported herein, indicate that interest in standardization in the textile field has been well maintained. The officers of the committee take this opportunity to express their full appreciation of the cooperative and valuable service rendered by the chairmen and members of the subcommittees during the past year, especially in view of the increased demands upon their time and energy imposed by war conditions.

The Advisory Committee has approved of the following changes in subcommittee and section chairmen: J. B. Goldberg as chairman of Subcommittee A-2, Section I on Rayon *vice* A. M. Tenney; E. M. Edgerton as chairman of Subcommittee A-2, Section II on Rayon Fabrics *vice* Alexis Sommaripa; E. M. Penney as chairman of Subcommittee A-8, Section I on Nomenclature and Definitions *vice* A. C. Clifford; J. F. Wooten as chairman of Subcommittee A-8, Section II on Yarn, Roving and Twine *vice* L. H. Nicholson. A. M. Tenney retains the general chairmanship of Subcommittee A-2 on Rayon and Its Products. K. H. Barnard is serving as acting chairman of Subcommittee B-4 on Bleaching, Dyeing and Finishing in place of W. M. Scott.

Society and committee appointments of representatives involving Committee D-13 members were as follows: W. D. Appel on Technical Committee X on Conditioning and Weathering of Committee E-1; A. G. Ashcroft and W. M. Scott on the Sectional Committee on Fastness of Colored Textiles (ASA Project: L 14); F. S. Mapes on the Sectional Committee on Electrical Insulating Materials (ASA Project: C 59) *vice* W. H. Bassett, Jr.; F. A. Prisley on the Standing Committee of Commercial Standard CS 65 – 38 on Wool and Part Wool Fabrics *vice* J. A. Levering; E. M. Penney on the Subcommittee on Definition of Glass of Committee E-8; A. G. Ashcroft to the hearing on April 10, 1942, by the Federal Trade Commission on color fastness of textiles.

The American Standards Association has approved as American standards two standards under the jurisdiction of Committee D-13, namely, Standard Specifications for Textile Testing Machines (D 76 – 41) and Standard Methods of Testing Wool Felt (D 461 – 40). The ASA designations which have been assigned to these standards are

L 15.1 – 1942 and L 16.1 – 1942, respectively.

In September, 1941, the Office of Price Administration requested the American Standards Association to draft definitions for denim and broadcloth. This request was referred to the American Society for Testing Materials and in turn to Committee D-13. A small, representative working committee was appointed with the approval of the ASA with A. G. Scroggie as its chairman. Under his skillful and wise guidance proposed definitions had been prepared and were ready for submittal to a larger group of interested parties when instructions were received from the OPA to discontinue the project.

The Membership Committee, G. E. Hopkins, chairman, has continued its quiet effective work throughout the year and is largely responsible for the steady growth of Committee D-13 which has brought it into first place in point of membership among the standing committees of the Society.

The present membership of Committee D-13 is 243; of whom 104 are classified as producers, 51 as consumers, and 88 as general interest members.

EMERGENCY ALTERNATE PROVISIONS IN STANDARDS

Emergency Alternate Provisions in the Standard Specifications and Methods of Test for Asbestos Roving for Electrical Purposes (D 375 – 41) waiving the magnetic iron requirements, were accepted by Committee E-10 on Standards on June 18, 1942, and have been issued under the designation EA – D 375.

RECOMMENDATIONS AFFECTING STANDARDS

Committee D-13 is presenting for publication as tentative two new specifications, six new methods of test for textile materials, and is recommending the revision of three tentative standards, the revision of five existing standards for immediate adoption, the tentative revision of two existing standards, the adoption as standard of six tentative standards and of six tentative revisions of existing standards, the withdrawal of one existing standard, and rhe reapproval of the following existing standards which have stood six years or more without revision:

Standard Specifications for:

Tolerances for Numbered Cotton Duck (D 230 – 27),

0.007-in. Cotton Tape for Electrical Purposes (D 335 – 36),

And Test for Holland Cloth (D 376 – 35),

Standard Methods of:

Testing and Tolerances for Certain Light and Medium Weight Cotton Fabrics (D 274 – 36),

Determining Relative Humidity (D 337 – 34), and

Test for Shrinkage in Laundering of Woven Cotton Cloth (D 437 – 36).

The revisions being presented are included in detail in Appendix I and are accompanied, where necessary, by brief statements of the reasons for such revisions. The new tentative standards and the existing standards affected are listed in Table I and are also referred to under subcommittee activities.

These recommendations have been submitted to letter ballot of the committee which consists of 243 members, the results being shown in Table I.

At the March, 1942, meeting the committee endorsed the American War Standard Specification and Description of Color (ASA Z44–1942) issued by the American Standards Association as an emergency standard and its use in connection with textiles. The committee also approved the publication of this emergency standard as an appendix to its annual compilation[1] for the information of its members.

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[1] See special compilation, "A.S.T.M. Standards on Textile Materials," 1942, p. 360; also ASTM BULLETIN, No. 119, December 1942, p. 20.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. New Tentative Standards			
Spec. for Asbestos Cloth	30	0	64
Spec. for Finished All-Cotton Upholstery Tapestries	33	0	61
Testing and Tolerances for Certain All-Cotton and Cotton-and-Rayon Fine Fancy Goods	40	1	53
Testing and Tolerances for Certain Fine Staple Gray Goods	43	1	50
Testing and Tolerances for Jute Rove and Plied Yarn for Electrical and Packing Purposes	26	0	68
Test for Colorfastness to Atmospheric Gases of Dyed Cellulose Acetate Rayon	41	0	53
Test for Identification of Finishes on Textiles	52	0	42
Resistance of Textile Fabrics to Microorganisms	43	2	49
II. Revisions of Tentative Standards			
Testing and Tolerances for Woven Tapes (D 259 - 39 T)	36	0	58
Testing and Tolerances for Certain Wool and Part Wool Fabrics (D 462 - 37 T)	47	1	46
Quantitative Analysis of Textiles (D 629 - 41 T)	57	1	36
III. Revisions of Standards, Immediate Adoption			
Testing and Tolerances for Hose Ducks and Belt Ducks (D 181 - 40)	30	2	62
Testing and Tolerances for Continuous Filament Rayon Yarns (D 258 - 40)	38	0	56
Testing Pile Floor Covering (D 418 - 40)	33	0	61
Spec. and Test for Asbestos Roving for Electrical Purposes (D 375 - 41)	26	0	68
Test for Fastness of Dyed or Printed Cotton Textiles to Laundering or Domestic Washing (D 435 - 41)	46	0	48
IV. Tentative Revisions of Standards			
Testing Wool Felt (D 461 - 40)	29	0	65
Spec. and Test for Fineness of Wool Tops (D 472 - 41)	34	1	59
V. Adoption of Tentative Standards as Standard			
Test for Commercial Weight of Continuous Filament Rayon Yarns (D 258 - 41 T), as revised	34	1	59
Test for Resistance to Yarn Slippage in Silk, Rayon, and Silk-Rayon Woven Fabrics (D 434 - 36 T)	40	0	54
Testing Rayon Staple (D 540 - 41 T)	36	0	58
Test for Commercial Weight of Spun Rayon Yarns and Threads (D 507 - 41 T), as revised	36	1	57
Spec. and Test for Asbestos Yarns (D 299 -40 T)	25	0	69
Testing Woven Asbestos Cloth (D 577 - 40 T)	27	0	67
VI. Adoption as Standard of Tentative Revisions of Standards			
Testing and Tolerances for Cotton Yarns (D 180 - 41)	49	1	44
Testing and Tolerances for Cotton Sewing Threads (D 204 - 39)	42	0	52
Testing and Tolerances for Tire Cord, Woven and on Cones (D 179 - 38)	40	2	52
Testing Pile Floor Covering (D 418 - 40)	34	0	60
Spec. for Textile Testing Machines (D 76 - 41)	68	0	26
Def. of Terms Relating to Textile Materials (D 123- 41)	67	1	26
VII. Withdrawal of Standard			
Test for Estimating Hard Scoured Wool in the Grease (Mill Scale Operations) (D 232 - 36)	30	0	64
VIII. Reaffirmation of Standards			
Spec. for Tolerances for Numbered Cotton Duck (D 230 - 27)	40	0	54
Spec. for 0.007-in. Cotton Tape for Electrical Purposes (D 335 - 36)	34	0	60
Spec. and Test for Holland Cloth (D 376 - 35)	36	0	58
Testing and Tolerances for Certain Light and Medium Weight Cotton Fabrics (D 274 - 36)	47	0	47
Determining Relative Humidity (D 337 - 34)	68	0	26
Test for Shrinkage in Laundering of Woven Cotton Cloth (D 437 - 36; ASA L10-1936)	46	1	47

The committee also approved for publication as information as an appendix to its annual report a Proposed Method of Test for Diffuse Transmission of Blackout Material for Incandescent-Lamp Light, taken from the proposed Amendment 3 to Federal Specification CCC-C-521a for Shade Cloth.

ACTIVITIES OF SUBCOMMITTEES

The recommendations being submitted indicate that the various subcommittees and sections have been quite active during the year. Many important projects are under consideration and are mentioned briefly in the following summary of subcommittee activities.

Subcommittee A-1 on Cotton and Its Products (K. B. Cook, chairman): *Section I on Cotton* (R. W. Webb, chairman) is recommending that the Tentative General Methods of Testing Cotton Fibers (D 414 - 40 T) be continued as tentative. The methods are being used and various points are being checked, but they are still considered in the experimental stage.

The section is studying new methods

for the measurement of length and strength of cotton fibers.

Section II on Cotton Yarns and Threads (A. C. Clifford, chairman) is recommending the adoption as standard of the tentative revisions[2] in the Standard General Methods of Testing and Tolerances for Cotton Yarns (D 180 – 41) on determination of twist and appearance of cotton yarns, and of the tentative revision[3] in the Standard Methods of Testing and Tolerances for Cotton Sewing Thread (D 204 – 39) on tolerance for yardage.

The section is studying methods of test for ball warps, determination of regain, twist in single yarn component of plied yarns, type of jaws for strength test of heavily plied yarns, tolerance for commercial regain, three methods for determination of twist in single yarns, tension in single yarn twist tests, and a number of tests for yarn number and strength determinations.

Section III on Light and Medium Weight Cotton Woven Fabrics (R. T. Fisher, chairman) is recommending for publication two new tentative standards: namely, proposed Tentative Methods of Testing and Tolerances for Certain All-Cotton and Cotton-and-Rayon Fine Fancy Goods,[4] and Methods of Testing and Tolerances for Certain Fine Staple Cotton Gray Goods.[4]

The section has reviewed the Standard Methods of Testing and Tolerances for Certain Light and Medium Weight Cotton Fabrics (D 274 – 36), and the Standard Method of Test for Shrinkage in Laundering of Woven Cotton Cloth (D 437 – 36), and finds them in accord with present practice and recommends their reapproval.

Section IV on Tire Fabrics (Martin Castricum, chairman) recommends the adoption as standard of the tentative revisions[5] in the Standard Methods of Testing and Tolerances for Tire Cord, Woven and on Cones (D 179 – 38) which enlarges the scope to include rayon cords, provides a sampling procedure, and specifies number of tests.

The section has reviewed the Standard Specifications and Methods of Test for Holland Cloth (D 376 – 35) and finds them in accord with present practice and recommends their reapproval.

Studies are being conducted on test conditions and methods of testing rayon cord.

Section V on Heavy Cotton Woven Fabrics (B. L. Whittier, chairman) recommends for immediate adoption, a revision of the Standard Methods of Testing and Tolerances for Hose Ducks and Belt Ducks (D 181 – 40) as appended hereto,[6] covering specifications for certain hose and belt ducks.

The section has reviewed the Standard Specifications for Tolerances for Numbered Cotton Duck (D 230 – 27) and finds them in accord with present practice and recommends their reapproval.

The section is studying specifications for ducks to be incorporated in Standard Specifications D 230 and is continuing its study of means to eliminate jaw breaks in testing heavy fabrics for strength.

Section VI on Narrow Fabrics (F. S. Mapes, chairman) recommends a revision of the Tentative Methods of Testing and Tolerances for Woven Tapes (D 259 – 39 T) to provide a breaking strength test in the direction of the filling, as given in Appendix I.

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[2] 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 636.

[3] 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 637.

[4] These methods were accepted as tentative by the Society and appear in the 1942 Book of A.S.T.M. Standards, Part III, pp. 1496 and 1494, respectively.

[5] 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 637.

[6] These revised methods were adopted as standard by the Society and appear in their latest revised form in the 1942 Book of A.S.T.M. Standards, Part III, p 583.

This section has reviewed the Standard Specifications for 0.007-in. Cotton Tape for Electrical Purposes (D 335 – 36) and finds them in accord with present practice and recommends their reapproval.

Subcommittee A-2 on Rayon and Its Products (A. M. Tenney, chairman):

Section I on Rayon (J. B. Goldberg, chairman) recommends, for immediate adoption, as given in Appendix I, a revision of the Standard Methods of Testing and Tolerances for Continuous Filament Rayon Yarns (D 258 – 40) to include provision for number of tests.

The section is recommending that the Tentative Method of Test for Commercial Weight of Continuous Filament Rayon Yarns (D 258 – 41 T), revised as given in Appendix I, be adopted as standard and incorporated in Standard Methods D 258 – 40.

The section is studying the number of tests required for determining denier and twist of rayon filament yarns.

Section II on Rayon Fabrics (E. M. Edgerton, chairman) recommends the adoption as standard of the Tentative Method of Test for Resistance to Yarn Slippage in Silk, Rayon, and Silk-Rayon Woven Fabrics (D 434 – 36 T).[7] This standard has been held tentative pending the investigation of shrinkage problems. No adverse criticisms have been received.

Section III on Rayon Staple and Spun Rayon Yarn (K. B. Cook, chairman) recommends the adoption as standard of Tentative Methods of Testing Rayon Staple (D 540 – 41 T)[8]; the section also recommends that the Tentative Method of Test for Commercial Weight of Spun Rayon Yarns and Threads (D 507 – 41 T), revised as given in Appendix I, be adopted as standard and incorporated in Standard Methods D 507 – 39.

This section is developing evenness standards for spun rayon yarns and methods of test for commercial weight and resiliency of rayon staple.

Subcommittee A-3 on Wool and Its Products (A. G. Ashcroft, chairman):

Section I on Wool (Werner von Bergen, chairman) recommends that the revision of the Standard Specifications and Methods of Test for Fineness of Wool Tops (D 472 – 41), published as tentative in 1941,[9] which provides for requirements or grades 48's to 36's, be further revised, as given in Appendix I, to bring the requirements in line with those for grades 80's to 50's.

The section is recommending the withdrawal of the Standard Method of Test for Estimating Hard Scoured Wool in Wool in the Grease (Mill Scale Operations) (D 232 – 36).[10] In conjunction with the wool trade and the Treasury Department a new method of estimating the clean content of wool in wool in the grease is being developed.

The section is conducting an interlaboratory study of fineness measurement of top, card sliver, and noils and of nep count in top.

Section II on Felt (G. H. Harnden, chairman) recommends for publication as tentative a revision of the Standard Methods of Testing Wool Felt (D 461 – 40) as given in Appendix I, to provide methods of test for water shrinkage and thickness swell.

The section is studying definitions, specifications, and methods of test for fiber content, acid determination, and fastness to soap and weather.

Section III on Woolen and Worsted Yarns (Fred Noechel, chairman) is studying the effect of tension in reeling on yarn number determination, and is conducting interlaboratory tests to determine the number of tests to be used for yarn number determinations.

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[7] 1939 Book of A.S.T.M. Standards, Part III, p. 1031.

[8] 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 565.

[9] *Ibid.*, p. 635.

[10] 1939 Book of A.S.T.M. Standards, Part III, p. 448.

Section IV on Pile Floor Coverings (J. N. Dow, chairman) recommends for immediate adoption a revision of the Standard Methods of Testing Pile Floor Covering (D 418 – 40) to include a section on precision of results for axminster carpets as given in Appendix I. This section is also recommending the adoption as standard of the tentative revision[11] of Standard Methods D 418 providing a form for reporting test results.

The section is continuing its studies on wear testing, mothproofing, and flameproofing.

Section V on Woolen and Worsted Fabrics (F. A. Prisley, chairman) recommends a revision of Tentative Methods of Testing and Tolerances for Certain Wool and Part Wool Fabrics (D 462 – 37 T) to bring the standard in accord with the Wool Products Labeling Act as appended hereto.[12]

The section will develop various test methods to evaluate serviceability and performance of fabrics under consumer conditions.

Subcommittee A-4 on Asbestos and Its Textile Products (F. S. Mapes, chairman):

This subcommittee submits for publication as tentative new proposed Tentative Specifications for Woven Asbestos Cloth.[13]

The subcommittee is recommending for immediate adoption a revision, of the Standard Specifications and Methods of Test for Asbestos Roving for Electrical Purposes (D 375 – 41) as given in Appendix I, covering an additional method (U. S. Navy method) for determination of total iron. The subcommittee has approved, as an emergency provision, the waiver of Section 5 on requirements for iron content and Section 17 (revised numbering) on method for determination of magnetic rating in Standard D 375.

The subcommittee also recommends the adoption as standard of the Tentative Specifications and Methods of Test for Asbestos Yarns (D 299 – 40 T)[14] and the Tentative Methods of Testing Woven Asbestos Cloth (D 577 – 40 T).[15]

The subcommittee is continuing its studies of the evaluation of grades of asbestos textiles on a time-temperature basis, and of methods for determining the relative value of asbestos textiles from an insulation standpoint.

Subcommittee A-5 on Bast and Leaf Fibers and Their Products (A. R. Howe, chairman):

This subcommittee submits for publication as tentative new proposed Tentative Methods of Testing and Tolerances for Jute Rove and Plied Yarns for Electrical and Packing Purposes.[16]

The subcommittee is developing test methods for flax and hemp products and has under consideration similar tests for jute twine, sisal and manila products. The classification of commercially important bast and leaf fibers with description, photographs, and chemical and microscopical characteristics is under consideration.

Subcommittee A-6 on Household and Garment Fabrics (Ruth O'Brien, chairman):

This subcommittee recommends for publication as tentative new proposed Tentative Specifications for Finished All-Cotton Upholstery Tapestries.[17]

The subcommittee recommends that the Tentative Specifications for Bleached

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[11] 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 638.

[12] These revised methods were accepted as tentative by the Society and appear in the in their latest revised form 1942 Book of A.S.T.M. Standards, Part III, p. 1526.

[13] These specifications were accepted as tentative by the Society and appear in the 1942 Book of A.S.T.M. Standards, Part III, p. 1455.

[14] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 430.

[15] *Ibid.*, p. 434.

[16] These methods were accepted as tentative by the Society and appear in the 1942 Book of A.S.T.M. Standards, Part III, p. 1515.

[17] These specifications were accepted as tentative by the Society and appear in the 1942 Book of A.S.T.M. Standards, Part III, p. 1465.

Wide Cotton Sheeting (D 503 – 40 T), and for All Wool, All Cotton, and Wool and Cotton Blanketing (Household) (D 576 – 40 T) be continued as tentative because of the abnormal situation regarding qualities of civilian goods brought about by the tremendous burden which military requirements have placed on the textile industry.

The subcommittee is conducting studies on the serviceability of sheeting and dishtoweling.

Subcommittee A-8 on Glass Fiber and Its Products (Dean Harvey, chairman):

This subcommittee recommends that the Tentative Methods of Testing and Tolerances for Glass Yarn (D 578 – 40 T) be continued as tentative pending the development of a method of test for yarn number, and also that the Tentative Methods of Testing and Tolerances for Woven Glass Tubular Sleeving and Braids (D 581 – 40 T) be continued as tentative pending more extensive practical experience in the use of these methods.

The subcommittee is studying contraction of yarn due to twist, determination of yarn number, abrasion methods, corrosion of copper by sizing, and compatibility of sizing.

Subcommittee B-1 on Methods and Machines (W. D. Appel, chairman):

Section I on Methods (W. D- Appel, chairman) recommends for publication new proposed Tentative Method of Test for Colorfastness to Atmospheric Gases of Dyed Cellulose Acetate Rayon.[18] This section is also recommending a revision of Tentative Methods of Quantitative Analysis of Textiles (D 629 – 41 T) as appended hereto.[19]

The section is continuing its studies on accelerated aging, identification of fibers, resilience of fibers, and soiling of textiles. The development of quantitative methods for various new fibers will be undertaken.

Section II on Machines (H. J. Ball, chairman) recommends the adoption as standard of the tentative revision[20] in Standard Specifications for Textile Testing Machines (D 76 – 41) which provides requirements for twist testers.

The section is continuing its studies on sensitivity of type A machines, effect of rate of loading on strength and elongation of cotton yarns, and specifications for yarn reels and drying ovens.

Section III on Atmospheric Conditions (R. H. Brown, chairman) has reviewed the Standard Method of Determining Relative Humidity (D 337 – 34) and finds it in accord with present practice and recommends its reapproval.

The section is studying, jointly with Section II, drying and conditioning technique. Regain data on specific materials are being collected for publication in the compilation of "A.S.T.M. Standards on Textile Materials."

Subcommittee B-2 on Nomenclature and Definitions (A. G. Scroggie, chairman):

This subcommittee recommends the adoption as standard of the tentative revision[20] in the Standard Definitions of Terms Relating to Textile Materials (D 123 – 41) of the term "Weight, Commercial" with an editorial revision to retain the word "moisture" now indicated to be omitted, and the term "Weight, Commercial (Rayon)" with an editorial change to add the word "moisture" before the word "regain." The definition of the term resilience is being continued as tentative.

Subcommittee B-4 on Bleaching, Dyeing and Finishing (W. M. Scott, chairman, K. H. Barnard, acting chairman):

This subcommittee is recommending for publication two new tentative standards: namely, Tentative Methods of Test for Identification of Finishes on Textiles,[21] and for Resistance of Textile Fabrics to Microorganisms.[21]

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[18] This method was accepted as tentative by the Society and appears in the 1942 Book of A.S.T.M. Standards, Part III, p. 1519.

[19] These revised methods were accepted as tentative by the Society and appear in the 1942 Book of A.S.T.M. Standards, Part III, p. 1425.

[20] 1941 Supplement to Book of A.S.T.M. Standards, Part III, pp. 635, 639.

The subcommittee is recommending for immediate adoption a revision of the Standard Method of Test for Fastness of Dyed or Printed Cotton Textiles to Laundering or Domestic Washing (D 435 – 41) to include linen and add two washing tests, one less severe and one more severe than the present tests. The revised Method of Test for Colorfastness to Commercial Laundering and to Domestic Washing of Cotton and Linen Textiles[22] has been prepared in cooperation with Sectional Committee L14 on Fastness of Colored Textiles, functioning under the joint sponsorship of the American Association of Textile Chemists and Colorists and A.S.T.M., under the procedure of the American Standards Association. The revised standard is based primarily upon the method recently developed by the Research Committee of the A.A.T.C.C.

The subcommittee is recommending that the Tentative Method of Test for Apparent Fluidity of Dispersions of Cellulose Fibers in Cuprammonium Hydroxide (D 539 – 40 T) be continued as tentative pending further consideration of a proposed revision.

The subcommittee is also recommending the publication as information of the American War Standard Specification and Description of Color (ASA Z44–1942)[1] and a Proposed Method of Test for Diffuse Transmission of Blackout Material for Incandescent-Lamp Light, the latter is given in Appendix II and has been taken from the proposed Amendment 3 to Federal Specification CCC-C-521a for Shade Cloth.

The subcommittee recommends that the Tentative Methods of Test for Resistance of Textile Fabrics to Water (D 583 – 40 T) be continued as tentative pending the formulation of contemplated revisions and that the Tentative Method of Test for Volumetric Determination of Small Amounts of Copper in Textiles (D 463 – 37 T) be continued as tentative pending the results of a study to determine the advisability of revising or withdrawing it.

This subcommittee is prepared to give prompt consideration to the additional methods of test for the fastness of colored textiles which are being revised to provide uniformity of procedure as ASA Project L14 under the joint sponsorship of A.S.T.M. and A.A.T.C.C., as well as to the special methods for the evaluation of hand of a fabric developed by the Special Committee for Testing Textile Finishes, as mentioned elsewhere in this report, as soon as they are received.

Subcommittee B-5 on Sampling, Presentation, and Interpretation of Data (A. G. Ashcroft, chairman):

This subcommittee has been active in assisting other subcommittees in making statistical studies of data with a view to establishing the number of tests required to give the desired precision. The subcommittee's Proposed Recommended Practice for Calculating Number of Tests to be Specified in Determining Average Quality of a Textile Material is published as information in the compilation of "A.S.T.M. Standards on Textile Materials."

The subcommittee sponsored a Symposium on Statistical Methods at the October, 1941, meeting. The titles of the symposium papers are given under the activities of Subcommittee B-6.

Subcommittee B-6 on Papers (R. H. Brown, chairman):

This subcommittee arranged for the

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[21] These methods were accepted as tentative by the Society and appear in the 1942 Book of A.S.T.M. Standards, Part III, pp. 1435 and 1444, respectively.

[22] This revised method was adopted as standard by the Society and appears in its latest revised form in the 1942 Book of A.S.T.M. Standards, Part III, p. 603.

presentation of the following papers at the October, 1941, meeting:

"Development of Methods for the Evaluation of Textile Finishes,"[23] by E. C. Dreby, as a report of the Special Committee for Testing of Textile Finishes.

"Engineering Use of Statistical Techniques in Testing,"[24] by A. E. Brandt,

"Application of the Statistical Method for Determining Number of Tests for Strength and Elongation of Rayon Yarns,"[24] by A. G. Scroggie,

"An Example of the Use of Statistics in a Study of Chemical Test Methods,"[24] by Lillian Weidenhammer,

"Application of Rank Correlation to the Development of Testing Methods,"[25] by E. R. Schwarz and K. R. Fox, and

"Report of Activities of Subcommittee A-8 on Glass Fiber," by K. N. Mathes.

The subcommittee also arranged for the presentation of the following paper at the March, 1942, meeting:

"Applications of A.S.T.M. Methods for the Manufacturing Control of French Spun Yarn," by Werner von Bergen and Fred Noechel.

Special Committee for Testing of Textile Finishes (M. C. Moore, chairman): The research project on the development of methods for the evaluation of finishes of textile fabrics was concluded on October 31, 1941, at the end of its second year. The following is a brief summary of what has been accomplished under this project:

1. A study has been made of the major properties of a fabric which go to make up the hand of that fabric. In cooperation with the A.A.T.C.C., these properties were listed, defined, and given considerable publicity. This should help to standardize the terminology, clarify thought, and eliminate confusion on the subject.

2. The three most important properties which go to make up the hand of a fabric were found to be flexibility, surface smoothness, and fullness.

3. Three instruments have been developed for measuring these properties together with another property, that of compressibility. These instruments are (*1*) the Friction Meter for measuring surface smoothness, (*2*) the Planoflex for measuring flexibility, and (*3*) the Compression Meter for measuring compressibility. It was found that a combination of the measurements made on the Planoflex and the Compression Meter gave a measure of fullness.

4. Realizing that these methods and instruments would be of value only in so far as they were accepted and used by the trade, a program of publicity has been organized including (*1*) a visit to all of the sponsors to demonstrate the instruments and methods, (*2*) a series of papers describing each of the instruments to be published shortly in technical journals, (*3*) and papers summarizing the project delivered by Mr. Dreby before Committee D-13 on October 16, 1942,[23] and before the Philadelphia Section of the A.A.T.C.C. on October 24, 1942.

A Permanent Committee has been appointed to administer the affairs of the Special Committee after the termination of the research program. This committee consists of George Slowinske, chairman, Laura E. Pratt, and W. D. Appel. The function of this committee is: first, to see to the adoption by Committee D-13 of the instruments and methods which have been developed; second, to answer inquiries which may arise after publication of papers describing the instruments and the methods for their use, and to promote the adoption of the instruments wherever possible; and third, to renew the research program at any time that the need of further study may be indicated.

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[23] Published in ASTM BULLETIN, No. 114, January, 1942, p. 15.

[24] Abstracts of these papers appear in the special compilation "A.S.T.M. Standards on Textile Materials," 1942, pp. 369, 372, and 376, respectively.

[25] Published in ASTM BULLETIN, No. 119, December, 1942, p. 21.

A Program Committee has been appointed to determine the interest of the subscribers in continuing the work into a third year. This committee consists of W. H. Steigler, chairman, F. Bonnet, and F. C. Scholler. The duty of this committee is to set up a program for a third year's work, provided a sufficient number of subscribers are interested in employing a research associate to take Mr. Dreby's place, and to raise sufficient funds to carry on the program. If a program is set up for a third year, the functions of the Permanent Committee will be turned over to an Administrative Committee for the third year program. In addition to further study of the application of the instruments which have been developed and possible further refinement of them, it might be found worthwhile in a third year program to do some work on the evaluation of crease-resistant finishes, shrink-resistant finishes and slip-resistant finishes. These are three items on the original program on which no work has been done as yet. There are now, however, available methods which have been considered reasonably satisfactory for evaluating them.

The following officers were elected at the March, 1942, meeting for the two-year period from June, 1942, to June, 1944:

Chairman, H. J. Ball.
First Vice-Chairman, F. S. Mapes.
Second Vice-Chairman, A. G. Ashcroft.
Secretary, W. H. Whitcomb.

This report has been submitted to letter ballot of the committee which consists of 243 members; 94 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. J. BALL,
Chairman.

W. H. WHITCOMB,
Secretary.

APPENDIX I

PROPOSED REVISIONS IN STANDARDS FOR TEXTILE MATERIALS

In this Appendix are given proposed revisions in certain standards and tentative standards covering textile materials which are referred to earlier in this report.[1] These standards and tentative standards appear in their present form either in the 1939 Book of A.S.T.M. Standards, Part III, or the 1940 and 1941 Supplements, Part III, as indicated by the final number in the A.S.T.M. designation.

REVISION OF TENTATIVE STANDARD

Tentative Methods of Testing and Tolerances for Woven Tapes (D 259 – 39 T):

Section 12.—Change from its present form to read as follows:

12. (*a*) The breaking strength of the tape shall be determined on a testing machine conforming to the Standard Specifications for Textile Testing Machines (A.S.T.M. Designation: D 76) of the American Society for Testing Materials. Test results, when specimens break in the clamps of the testing machine, shall be discarded.

(*b*) The specimens in the direction of the warp shall be tested in full section and the initial distance between clamps shall be 3 in.

(*c*) The specimens in the direction of the filling shall be tested by the grab method in accordance with Section 10 of Standard Methods D 39 and the initial distance between the clamps shall be ⅛ in.

(*d*) At least five determinations of the breaking strength shall be made in each direction and the average of these five tests shall indicate the strength of the roll.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

Standard Methods of Testing and Tolerances for Continuous Filament Rayon Yarns (D 258– 40):

Section 4.—Change from its present form to read as follows:

4. *Strength.*—The strength of the rayon yarn shall be expressed as tensile strength (grams per denier of the yarn before test) at the yield point or at rupture.

Section 19.—Add new Paragraphs (*d*), (*e*), and (*f*) to read as follows:

(*d*) The specimens of yarns for strength determination shall be drawn from the side of the package instead of over the top to avoid change in twist.

(*e*) Tests in which the specimen breaks at the jaw shall be rejected.

(*f*) The tensile strength shall be expressed in grams per denier by dividing the breaking strength by the denier of the yarn.

Section 20.—Change from its present form: namely,

20. Five single strands from each of 10 spools, cops, tubes, cones, or skeins shall be broken. The average of 50 tests free from breaks at the clamps shall be the breaking strength of the yarn. The tensile strength shall be determined by dividing the average breaking strength by the denier of the yarn.

to read as follows:

20. The number of tests, n, required for the determination of breaking strength and percentage elongation shall be normally 30, which value is based on the following conditions: Probability, 0.95; precision (random sampling error), E, 2.5 per cent; coefficient of variation, V, 7 per cent. The tests shall be distributed

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[1] See p. 424.

over 10 spools, cops, tubes, cones or skeins so that $\frac{n}{10}$ tests are made on each package.

NOTE.—Available information indicates that the value of the coefficient of variation (7 per cent) is higher than that of yarn currently supplied to the trade. If, at any time, the running level of V based on experience is found to be V_1, (greater than 7 per cent) the number of tests required shall be determined from the following formula:

$$n_1 = \frac{V_1^2 \times 30}{V^2}$$

The calculated value, n_1, shall be rounded off to the next higher multiple of 10. In no case shall n_1 be less than 30. This requirement prescribes 40 tests when V_1 falls between 7 and 8 per cent, for 50 tests when V_1 falls between 8 and 9 per cent, etc.

Standard Methods of Testing Pile Floor Covering (D 418 – 40):

Section 17 (a) and Table I.—Change to read as follows by the addition of the italicized words:

17. Precision of Results, Velvet *and Axminster* Carpets.—(*a*) The true coefficients of variation for each of the physical characteristics of velvet *and axminster* carpets are approximately those shown in Table I. The values in Table I may be used in estimating the precision of such measurements.

TABLE I.—VALUES OF v AND E FOR VELVET *AND AXMINSTER* CARPET.[a]

Characteristic	Coefficient of Variation, v, per cent		Limit of Uncertainty for One Determination, E, per cent (Chances of the order of 9 in 10)	
	Velvet	*Axminster*	*Velvet*	*Axminster*
Pile yarn weight	4.5	*4.5*	±7.5	*±7.5*
Total weight	3.0	*3.5*	±5.0	*±6.0*
Tuft length	2.0	*2.5*	±3.0	*±4.0*
Total thickness	2.5	*3.0*	±4.5	*±5.0*
Net pile thickness	3.5	*5.0*	±5.5	*±8.0*

[a] The information in this table is based upon data obtained from a large number of analyses on typical velvet *and axminster* grades made by three manufacturers.

Standard Specifications and Methods of Test for Asbestos Roving for Electrical Purposes (D 375 - 41):

New Sections.—Add the following new Sections 14 to 16 comprising an alternative procedure for determining total iron, renumbering the subsequent sections accordingly. Designate the present procedure as "Method A" and the new procedure as "Method B."

METHOD B

14. *Reagents: (a) Phenanthroline Indicator.*—The solution may be purchased ready for use.

(*b*) *Hydrochloric Acid (1:10).*—Mix 1 volume of HCl (sp. gr. 1.19) and 10 volumes of distilled water. Each specimen will require 100 ml. of this solution.

(*c*) *Ceric Ammonium Sulfate Solution (0.1 N).*—Dissolve 80 g. of ceric ammonium sulfate ($Ce(SO_4)_2 \cdot 2(NH_4)_2SO_4 \cdot 2H_2O$) in 500 ml. of a H_2SO_4 solution (prepared by diluting 28 ml. of H_2SO_4 (sp. gr. 1.84) with distilled water), stir until dissolved, and then dilute to 1000 ml. Standardize the solution, which is approximately 0.1 N, against a solution containing a known amount of iron in accordance with the procedure described in Section 15 (*b*).

15. *Procedure: (a) Ash.*—Dry a sample of approximately 1 g. to constant weight in an oven at 105 to 110 C., transfer the specimen to a desiccator, allow to cool to room temperature, and then reweigh. This is the oven-dry weight of the specimen, weight D. Place the specimen in a tared No. 0 porcelain crucible and ignite in a muffle furnace at approximately 900 C. for 20 to 30 min. Transfer the crucible and specimen to a desiccator, cool to room temperature, and then reweigh. The difference between this weight and the tare weight of the crucible is the weight of ash, weight A. The amount of ash shall be calculated as a percentage of the oven-dry weight of the specimen, as follows:

$$\text{Ash, per cent} = \frac{A}{D} \times 100$$

(*b*) *Total Iron.*—Transfer the ash (Paragraph (*a*)) to a 300-ml. glass beaker, add 25 ml. of c.p. HCl (sp. gr. 1.19), and heat to boiling. While the solution is boiling, add just a sufficient number of drops of c.p. HF to completely dissolve the iron, and then concentrate by boiling to a volume of 15 ml., cool, and dilute with 100 ml. of cold distilled water. Pass the solution through a Walden reductor, wash the reductor with 100 ml. of diluted HCl (1:10), add 15 to 20 ml. of H_2SO_4 (sp. gr. 1.84) and 3 drops of phenanthroline indicator to the filtrate, and then titrate with the standard ceric ammonium sulfate.

16. *Calculation and Report.*—Report the total iron as a percentage of the oven-dry weight of the specimen as calculated from the following formula:

$$\text{Total iron, per cent} = \frac{M \times N \times 0.05584}{D} \times 100$$

where:

M = milliliters of the standard ceric ammonium sulfate solution,

N = normality of the ceric ammonium sulfate solution, and

D = oven-dry weight of the specimen.

TENTATIVE REVISIONS OF STANDARDS

Standard Methods of Testing Wool Felt (D 461 - 40):

Section 1.—Designate this section as Paragraph (*a*) and add a new Paragraph (*b*) to read as follows:

(*b*) The procedure for physical tests and chemical analysis as described in Section 4 to 14 are applicable to all felts as described in Paragraph (*a*). The special test procedures described in Sections 15 to 17 are applicable only to the testing of felts for certain special applications and only when specified in the purchase order or material specifications.

Tentative revision, submitted June, 1942.

New Sections.—Add new Sections 15 to 17 to read as follows:

SPECIAL TESTS

Roll Wool Felt

15. *Water Shrinkage:* (*a*) *Sampling.*—Two samples, each approximately 12 by 12 in. shall be cut, preferably back from the end of the felt a distance equal to the width of the piece (Note 1). One sample shall be cut midway between the edges (marked as sample C) and the other shall be cut from an outer edge, inside of any pin holes (marked as sample E). Each sample shall be legibly marked with indelible ink or by other means to indicate the lengthwise and widthwise direction in the original piece.

NOTE 1.—If for any reason it is impractical to cut samples from the locations specified, they may be cut from the outer end of the felt. In such cases, samples should be taken from two different rolls and the test results averaged.

(*b*) *Test Specimens.*—From each sample a test specimen shall be cut having minimum dimensions of 10 by 10 in,, using a template of suitable size to insure parallel sides. Each test specimen shall bear the identification marks prescribed in Paragraph (*a*).

(*c*) *Procedure.*—The width and length of each test specimen shall be accurately measured with a steel scale. The thickness of each specimen shall be determined by taking the average of 12 measurements (three on each side) with a dial micrometer as described in Section 6 of the Standard General Methods of Testing Woven Textile Fabrics (A.S.T.M. Designation: D 39) of the American Society for Testing Materials. The specimens shall then be placed on a wire screen (preferably with $\frac{1}{4}$-in. openings) and immersed in boiling water for 2 min. (Note 2). The screen and specimens shall then be withdrawn, the specimens removed, and placed in a flat position between blotting paper to remove excess moisture, care being taken not to distort the wet specimen in either direction. Squeeze rolls shall not be used to remove further moisture. As soon as practicable, the width and and length of each test specimen shall be accurately measured to the nearest $\frac{1}{16}$ in., the measurements being made at the center and each side in both directions. The average of the three readings each of the width and length shall be recorded.

NOTE 2.—The use of a wetting agent is recommended for hard felts which will not wet out thoroughly in boiling water in 2 min.

(*d*) *Calculation.*—The water shrinkage of the specimens shall be calculated as the percentage loss in size from the original dimensions, as follows:

$$\text{Shrinkage in length, per cent} = \frac{L_1 - L_2}{L_1} \times 100$$

$$\text{Shrinkage in width, per cent} = \frac{W_1 - W_2}{W_1} \times 100$$

where:

L_1 = average length of the original specimens C and E,

L_2 = average length of specimens C and E after removal of excess moisture,

W_1 = average width of the original specimens C and E, and

W_2 = average width of specimens C and E after removal of excess moisture.

16. *Water Thickness Swell:* (*a*) *Procedure.*—The wet specimens obtained under Section 15 (*c*) shall be dried in an oven to constant weight at 105 to 110 C. Twelve thickness measure-

ments shall be made in accordance with the procedure described in Section 15 (*c*), and the average thickness calculated.

(*b*) *Calculation.*—The water thickness swell shall be calculated as the percentage increase in thickness from that of the original specimen, as follows:

$$\text{Thickness swell, per cent} = \frac{T_2 - T_1}{T_1} \times 100$$

where:

T_1 = average thickness of original specimen (Section 15 (*c*)), and

T_2 = average thickness of specimen after wetting and drying.

Sheet Wool Felt

17. *Water Thickness Swell:* Due to construction, no noticeable shrinkage can ordinarily be be measured on small specimens of sheet felts. While this test is primarily intended as a thickness swell test, the procedure described in Section 15 (*c*) can be used when a shrinkage test for felt is desired. In this case, the 10 by 10-in. specimens shall be used as described in Section 15 (*b*) and the specimens shall be cut with the outer edges at a 90 deg. angle.

(*a*) *Test Specimens.*—Two test specimens, each approximately 2 by 2 in. shall be cut from different portions of the sheet.

(*b*) *Procedure.*—The thickness of the test specimens shall be accurately measured with a dial micrometer as described in Section 6 of Standard Methods D 39. The specimens shall be placed on a wire screen and immersed in boiling water for 2 min. (Note 2, Section 15 (*c*)) as described in Section 15 (*c*). The specimens shall then be removed, and dried to constant weight at 105 to 110 C. At least four thickness measurements shall be made on each specimen and the average recorded.

(*c*) *Calculation.*—The percentage water thickness swell shall be calculated as described in Section 16 (*b*).

Standard Specifications and Methods of Test for Fineness of Wool Tops (D 472 - 41):

Table I.—In place of the requirements proposed as a tentative revision[2] in 1941, substitute the requirements for wool tops of grades 48's to 36's prescribed in the accompanying Table II.

TABLE II.—REQUIREMENTS FOR WOOL TOPS.

Grade	48's	46's	44's	40's	36's
Fineness Range, microns:					
Min.	31.6	33.3	34.8	36.6	38.8
Max.	33.2	34.7	36.5	38.7	41.3
Fibers, per cent:					
10 to 20 microns, incl., min.	....	....	....	....	....
10 to 25 microns, incl., min.	....	....	....	....	....
10 to 30 microns, incl., min.	34	26	22	16	12
10 to 40 microns, incl., min.	77	70	63	55	44
25.1 to 30 microns, incl., max.	....	....	....	....	....
25.1 to 40 microns, incl., max.	....	....	....	....	....
30.1 to 40 microns, incl., max.	....	....	....	....	....
30.1 to 50 microns, incl., max.	....	....	....	....	....
40.1 to 50 microns, incl., max.	....	....	....	....	....
40.1 to 70 microns, incl., max.	23	30	37	45	56
60.1 to 80 microns, incl., max.	2	2	3	4	4
Minimum number of fibers required for test, per sample	1600	1600	1600	1600	1600

ADOPTION OF TENTATIVE STANDARDS AS STANDARD

Tentative Method of Test for Commercial Weight of Continuous Filament Rayon Yarns (D 258 - 41 T):

Section 7.—Change Paragraph (*b*) to read as follows by the addition of the italicized words and figure and the omission of those in brackets:

> (*b*) Bath.—The weight of the boil-off bath shall be 25 times the combined weight of the two test specimens and shall contain [0.25 g. of sulfated fatty alcohol detergent] *0.5 g. of neutral soap* per liter.

Omit footnote 3 which reads as follows:

> Gardinol LS flakes or their equivalent are satisfactory for this purpose.

Change the first sentence of Paragraph (*c*) to read as follows by the addition of the italicized words:

> The two test specimens shall be treated simultaneously for 30 min. at the boiling temperature in the case of viscose or cuprammonium rayon; or at 71 to 77 C. in the case of acetate rayon, *or mixtures containing acetate rayon.*

Section 8.—Change the last sentence to read as follows by the omission of the words in brackets:

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[2] 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 635.

The container shall then be closed, removed from the oven, and allowed to cool [in a dry atmosphere] before weighing.

Tentative Method of Test for Commercial Weight of Spun Rayon Yarns and Threads (D 507 – 41 T):

Sections 7 and 8.—Make the same changes in these sections as recommended above in Sections 7 and 8 of Methods D 258 – 41 T.

APPENDIX II

PROPOSED METHOD OF TEST FOR DIFFUSE TRANSMISSION OF BLACKOUT MATERIAL FOR INCANDESCENT LAMP LIGHT[1]

1. (*a*) The diffuse luminous transmission of a single layer of blackout material shall be obtained by placing it in front of a bright diffusing source and by comparing (with any suitable means) the brightness of the unlighted side of the material with the brightness of the source observed directly. For the source, a white-lined inclosure may be used, with a projection-type incandescent lamp inside and a shield so placed as to hide the filament of the lamp from direct view through the opening of the inclosure. As an alternate source for dark materials, one may use a diffusing screen, such as a piece of white opal glass, with an incandescent lamp of high intensity behind it. In either case, the illumination of the test specimen shall be sufficient to permit brightness settings to be made for specimens of diffuse luminous transmission equal to 0.001 of 1 per cent, and the lamp shall operate at a color temperature of 2800 to 3200° Kelvin.

(*b*) Suitable instruments with which to compare the brightness of the lighted blackout materials with the brightness of the source used behind them are: (*1*) the Macbeth Illuminometer (sold by Leeds and Northrup Co., 4901 Stenton Ave., Philadelphia, Pa.), (*2*) the Luckiesh-Taylor brightness meter (sold by the General Electric Co., Nela Park, Cleveland, O.), and (*3*) the Holophane Lightmeter (sold by the Holophane Glass Co., 342 Madison Ave., New York, N. Y.). For these instruments an illumination of about 10,000 foot candles on the test specimen has been found to be suitable. Photoelectric devices may be used when it is known that they give equivalent (luminous) values; however, many photoelectric cells are unsuitable for this purpose because of their response to infrared radiation.

(*c*) At least three separate areas of each sample shall be measured. Observation shall be from the direction in which the sample appears brightest (this is usually the perpendicular direction).

2. *Opacity.*—When specified "for blackout purposes," the diffuse luminous transmission of a single layer of the shade cloth of any type shall average no more than 0.001 of 1 per cent; no area of the shade cloth shall transmit more than 0.003 of 1 per cent; and the material shall have no large pin holes or streaks which transmit an appreciable amount of light.

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[1] Reproduced from Amendment 3, March 23, 1942, to Federal Specification for Shade Cloth (CCC-C-521a).

REPORT OF SECTIONAL COMMITTEE
ON
FASTNESS OF COLORED TEXTILES
ASA PROJECT: L14

Sectional Committee L14 on Fastness of Colored Textiles was organized in April, 1941, under the procedure of the American Standards Association with the American Association of Textile Chemists and Colorists and the American Society for Testing Materials as co-sponsors. The scope of the committee is as follows:

Scope.—Correlation of standards covering test methods for the fastness of colored textiles to color-destroying agencies and the evaluation and interpretation of data obtained from such tests—the work to be based upon standards developed by cooperating organizations.

The personnel of the committee was approved by the ASA on May 21, 1942, and includes representatives from the following organizations:

American Association of Textile Chemists and Colorists,
American Association of Textile Technologists,
American Association of University Women,
American Council of Commercial Laboratories,
American Home Economics Assn.,
American Institute of Laundering,
American Society for Testing Materials,
Cotton-Textile Institute,
Limited Price Variety Stores Assn.,
Mail Order Group,
National Assn. of Dyers and Cleaners,
National Assn. of Finishers of Textile Fabrics,
National Assn. of Wool Manufacturers,
National Bureau of Standards,
National Federation of Textiles, Inc.,
National Retail Dry Goods Assn.,
Synthetic Organic Chemical Manufacturers Assn.,
Textile Fabrics Assn.,
U. S. Department of Agriculture, and
Office of Price Administration (*liaison*).

Meetings of the committee were held in New York, N. Y., on July 9, 1941, February 17, 1942, and on March 14, 1942. Several meetings of each of the two subcommittees have been held.

The subcommittee interested in the correlation of standard test methods has prepared for consideration as an American standard a Method of Test for Colorfastness to Commercial Laundering and to Domestic Washing of Cotton and Linen Textiles.[1] This work was done under the chairmanship of W. D. Appel and is a correlation of the test methods described in the A.A.T.C.C. Year Book,[2] in A.S.T.M. Standard Method D 435 - 41,[3] and in the Commercial Standard for Woven Textile Fabrics, Testing and Reporting (CS59-41). Similar efforts at correlation are being made for the other test methods for determining the colorfastness of fabrics.

The subcommittee on terminology to describe fastness of colored fabrics to color-destroying agencies was expedited by a request, dated February 7, 1942, from the Office of Price Administration, to develop a system of terminology to assist the Federal Trade Commission. Recommendations made by this subcommittee were acted upon by Sectional

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[1] See report of Committee D-13 on Textile Materials, p. 430.

[2] Fastness Tests for Dyed or Printed Cotton, 1941 Year Book, Am. Assn. Textile Chemists and Colorists, Vol. XVIII, p. 186.

[3] Standard Method of Test for Fastness of Dyed or Printed Cotton Textiles to Laundering or Domestic Washing (D 435 - 41), 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 163.

Committee L14 at its meetings in February and March and were then transmitted to the O.P.A. and the F.T.C. on April 7, 1942. These recommendations have not yet been submitted to letter ballot of the sectional committee inasmuch as they were intended to be of assistance to the F.T.C. at its hearing in New York on April 10, 1942, and the terminology adopted by the Commission would be the basis for the final American standard for the description of colorfastness of colored fabrics.

Respectfully submitted on behalf of the sectional committee,

JULES LABARTHE, JR.
Chairman.

B. A. RYBERG,
Secretary.

REPORT OF COMMITTEE D-17

ON

NAVAL STORES

A meeting of Committee D-17 on Naval Stores was held jointly with Subcommittee XII on Turpentine, of Committee D-1 on Paint, Varnish, Lacquer, and Related Products, on June 25, 1941, during the annual meeting of the Society in Chicago, Ill., at which time the work of the committee was discussed. Another meeting of the committee was held at Atlantic City, N. J., on June 23, 1942, during the 1942 Annual Meeting of the Society. The following report covers the activities of the committee and its subcommittees at both meetings.

ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the Tentative Methods of Test for Saponification Number of Rosin (D 464 – 37 T)[1] and for Acid Number of Rosin (D 465 – 37 T)[1] be approved for reference to letter ballot of the Society for adoption as standard with the inclusion of a limiting statement that these methods are not recommended for rosins of colors darker than grade G as indicated below:

Section 1.—Add the following as a note at the end of the section on Scope:

NOTE.—Due to difficulty in observing the end point in the titration, because of the dark color of the solution, this method using an internal indicator is not recommended for use with rosins of grades darker than G, that is, of grades D, E, F, and FF.

This recommendation has been submitted to letter ballot of the committee which consists of 20 members, 17 members returned their ballots, of whom 16 voted affirmatively, 0 negatively, and 1 member marked his ballot "not voting."

Work will be continued in order to obtain reliable methods for determining the acid and saponification numbers of the darker grades of rosin in which the end points of titration are difficult to distinguish. Electrometric titration methods will also be considered.

The activities of Subcommittee I on the Softening Point of Rosin (C. E. Kinney, chairman) and Subcommittee IV on Acid and Saponification Numbers and Unsaponifiable Matter (C. C. Zeigler, chairman) are covered in the appended reports.

The committee, as a result of the work of Subcommittee I, reported to Committee E-1 on Methods of Testing that the tapered ring and the shouldered ring as used in the Tentative Method of Test for Softening Point (Ball and Tapered Ring Apparatus) (E 28 – 39 T) can be considered interchangeable so far as rosin and similar resins are concerned.[2] In its report the committee also recommended to Committee E-1 that the use of a stirring device in the heating medium be required to ensure uniform heating, and that for samples softening around 80 C. (where either water or glycerine may have been used), the results of tests of softening point should carry a notation showing the nature of the bath fluid.

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[1] 1939 Book of A.S.T.M. Standards, Part II, pp 1145, 1144; Part III, pp. 1094, 1093.

[2] See Editorial Note, p. 482.

Since all members of this committee undoubtedly are now working under an unusual pressure due to the war program, each member was requested to give careful consideration to the work of all the subcommittees with the view of making recommendations as to which activities should be continued and which may be temporarily suspended.

A complete reorganization has been made in the set-up of subcommittees of Committee D-17. The new arrangement is indicated below:

Subcommittee I on Softening Point of Rosin
Subcommittee II on Crystallization of Rosin
Subcommittee III on Volatile Oils in Rosin
Subcommittee IV on Acid and Saponification Numbers and Unsaponifiable Matter
Subcommittee V on Specifications for and Analysis of Tall Oil (Liquid Rosin)
Subcommittee VI on Specifications for and Testing of Pine Tar and Tar Oil
Subcommittee VII on Terpenes Other than Turpentine

The Tentative Methods of Sampling and Grading Rosin (D 509 – 38 T) are being continued as tentative. Consideration is being given to the frequency of sampling rosin for grading where the rosin is produced by a continuous distillation process, since the recognized methods of sampling are all based on the batch production method.

The Standard Method of Test for Toluene Insoluble Solid Matter in Rosin (Chiefly Sand, Chips, Dirt, and Bark) (D 269 – 30) was reviewed in 1939 which resulted in several editorial improvements being incorporated in the method. No further changes are contemplated at this time.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, V. E. Grotlisch.
Vice-Chairman, E. V. Romaine.
Secretary, W. A. Kirklin.

This report has been submitted to letter ballot of the committee, which consists of 20 members; 20 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

F. P. VEITCH,
Chairman.

C. F. SPEH,
Secretary.

REPORT OF SUBCOMMITTEE I ON SOFTENING POINT OF ROSIN

Subcommittee I on Softening Point of Rosin has completed collaborative softening point tests on referee samples of rosins and resins with tapered and shouldered rings. The object of the tests was to determine the general applicability of the shouldered ring to the ring and ball method of test for softening point of rosins and resins and to determine its interchangeability, as redesigned, with the tapered ring specified in the Tentative Method of Test for Softening Point (Ball and Tapered Ring Apparatus) (E 28 – 39 T).[1]

The dimensions of the original shouldered ring, which was made the subject of collaborative tests on rosins and resins in 1939,[2] and the redesigned shouldered ring which was used in these collaborative tests are:

	Original Shouldered Ring of 1939 Tests	Redesigned Shouldered Ring of 1941–42 Tests
Depth:		
Inside top	9/64 in.	9/64 in.
Inside bottom	7/64 in.	7/64 in.
Outside top	5/32 in.	11/64 in.
Outside bottom	3/32 in.	5/64 in.
Diameter:		
Inside top	23/32 in.	25/32 in.
Inside bottom	41/64 in.	5/8 in.
Outside top	13/16 in.	29/32 in.
Outside bottom	3/4 in.	3/4 in.
Inside angle of shoulder	not rounded	rounded

Tentative Method E 28 – 39 T was followed in the 1941–42 tests except that stirring of the bath and heating of the bath with a hot plate were specified. However, some collaborators found it necessary to use a gas burner due to the failure of available hot plates with sufficient heating capacity to increase the temperature of the bath at the prescribed rate. A glycerol bath was used by all collaborators for the two samples with softening points above 80 C.

Table I contains individual determinations with average and maximum deviation figures for each collaboratoring laboratory and also a summary of these data which indicates the greatest probable deviation between the results obtained by the tapered and shouldered rings among individual laboratories. There is also included in Table I a summary giving the average of all individual laboratory averages, the maximum deviation from this average of averages, and the probable agreement of results which can be obtained with the two rings as calculated from the average values.

The results of the collaborative tests indicate that the shouldered ring can be used interchangeably with the present tapered ring. Other factors apparently exert a much greater effect on the softening point than the differences in ring design.

Respectfully submitted on behalf of the subcommittee,

C. E. Kinney,
Chairman.

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[1] 1939 Book of A.S.T.M. Standards, Part II, p. 1169, and Part III, p. 1127.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 420 1940).

TABLE I.—RESULTS OF SOFTENING POINT DETERMINATIONS ON REFEREE SAMPLES OF RESINS BY COLLABORATING LABORATORIES.

	WG Wood Rosin		FF Wood Rosin		Helix Wood Rosin		WW Gum Rosin		M Gum Rosin		Synthetic Resin	
	Tapered Ring	Shouldered Ring	Tapered Ring	Shouldered Ring	Tapered Ring	Shouldered Ring	Tapered Ring	Shouldered Ring	Tapered Ring	Shouldered Ring	Tapered Ring	Shouldered Ring
W. H. GARDNER, POLYTECHNIC INST. OF BROOKLYN.												
Individual determinations	75.30	76.30	71.20	72.20	135.90	136.90	70.70	71.90	74.70	75.90	144.20	144.90
	75.60	76.40	71.60	72.30	136.10	137.40	70.80	71.60	74.50	75.30	144.00	144.50
	75.30	76.60	71.70	72.40	136.30	137.00	70.80	71.80	74.80	75.80	144.60	144.30
	75.20	76.20	71.40	72.10	136.00	137.00	71.00	71.40	74.80	75.40	143.60	144.80
Average	75.40	76.40	71.50	72.30	136.10	137.10	70.80	71.70	74.70	75.60	144.10	144.60
Max. deviation	0.40	0.40	0.50	0.30	0.40	0.50	0.30	0.50	0.30	0.60	1.00	0.60
Max. deviation from average	+0.20	+0.20	+0.20	+0.10	+0.20	+0.30	+0.20	+0.20	+0.10	+0.30	+0.50	+0.30
	−0.20	−0.20	−0.20	−0.20	−0.20	−0.20	−0.10	−0.30	−0.20	−0.30	−0.50	−0.30
Average deviation of shouldered from tapered	+1.00		+0.80		+1.00		+0.90		+0.90		+0.50	
JOHN McE. SANDERSON, AMERICAN CYANAMID AND CHEMICAL CORP.												
Individual determinations	74.00	74.20	71.80	72.00	136.00	136.50	71.40	71.40	76.80	77.00	143.50	142.50
	74.00	74.40	72.00	72.00	136.50	136.00	71.60	71.20	76.80	76.80	143.00	143.00
	73.80	74.20	72.00	72.00	136.50	137.00	71.40	71.40	77.00	76.80	144.50	145.00
	74.00	74.20	71.80	71.80	136.00	136.50	71.40	71.40	77.00	76.80	145.00	145.00
Average	74.00	74.30	71.90	72.00	136.30	136.50	71.50	71.40	76.90	76.90	144.00	143.90
Max. deviation	0.20	0.20	0.20	0.20	0.50	1.00	0.20	0.20	0.20	0.20	2.00	2.50
Max. deviation from average	+0.00	+0.10	+0.10	+0.00	+0.20	+0.50	+0.10	+0.00	+0.10	+0.10	+1.00	+1.10
	−0.20	−0.10	−0.10	−0.20	−0.30	−0.50	−0.10	−0.20	−0.10	−0.10	−1.00	−1.40
Average deviation of shouldered from tapered	+0.30		+0.10		+0.20		−0.10		0.00		−0.10	
C. F. SPEH (W. C. SMITH) NAVAL STORES RESEARCH DIV. BUREAU OF AGRICULTURAL CHEMISTRY AND ENGINEERING, U. S. DEPT. OF AGRICULTURE												
Individual determinations	75.00	75.00	73.00	73.00	139.00	139.75	74.25	74.00	77.00	77.50	144.00	144.50
	74.75	74.75	73.50	73.50	139.25	140.00	74.00	74.25	77.00	77.75	144.00	144.50
	75.00	75.00	73.00	73.50	139.75	140.25	74.00	74.00	77.00	77.50	143.50	144.00
Average	74.90	74.90	73.20	73.30	139.30	140.00	74.10	74.10	77.00	77.60	143.80	144.30
Max. deviation	0.25	0.25	0.50	0.50	0.25	0.50	0.25	0.25	0.00	0.25	0.50	0.50
Max. deviation from average	+0.10	+0.10	+0.30	+0.20	+0.45	+0.25	+0.15	+0.15	+0.00	+0.15	+0.20	+0.20
	−0.15	−0.15	−0.20	−0.30	−0.30	−0.25	−0.10	−0.10	−0.00	−0.10	−0.30	−0.30
Average deviation of shouldered from tapered	0.00		+0.10		−0.70		0.00		+0.60		+0.50	
G. A. HAWKINS (D. MOWAT) SOUTHERN PINE CHEMICAL CO.												
Individual determinations	74.00	74.00	72.20	72.00	136.00	137.50	71.30	71.20	75.30	75.20	142.50	143.50
	74.30	74.20	72.40	72.10	136.00	137.50	71.40	71.40	75.40	75.30	142.50	143.40
	74.20	74.00	72.00	71.80	136.70	137.70	71.30	71.20	75.40	75.30	142.00	143.00
	74.50	74.20	72.10	72.00	136.30	137.30	71.30	71.40	75.40	75.40	142.50	143.20
Average	74.30	74.10	72.20	72.00	136.30	137.50	71.30	71.30	75.40	75.30	142.40	143.30
Max. deviation	0.50	0.20	0.40	0.30	0.70	0.40	0.10	0.20	0.10	0.20	0.50	0.50
Max. deviation from average	+0.20	+0.10	+0.20	+0.10	+0.40	+0.20	+0.10	+0.10	+0.00	+0.10	+0.10	+0.20
	−0.30	−0.10	−0.20	−0.20	−0.30	−0.20	−0.00	−0.10	−0.10	−0.10	−0.40	−0.30
Average deviation of shouldered from tapered	−0.20		−0.20		+1.20		0.00		−0.10		+0.90	
H. R. WOERNER (H. W. SHADER), ARMSTRONG CORK CO.												
Individual determinations	72.40	73.40	72.50	70.70	....	....	71.40	70.40	77.40	75.60	....	....
	72.80	73.50	72.60	70.90	....	....	72.00	71.00	77.70	75.80	....	....
	73.40	72.60	72.10	70.30	....	....	72.10	70.50	78.40	75.70	....	....
	73.60	72.90	72.50	70.90	....	....	72.30	71.20	78.80	75.90	....	....
Average	73.00	73.10	72.40	70.70	....	....	72.00	70.80	78.10	75.80	....	....
Max. deviation	1.20	0.90	0.50	0.60	....	....	0.90	0.80	1.40	0.30	....	....
Max. deviation from average	+0.60	+0.40	+0.20	+0.20	....	....	+0.30	+0.40	+0.70	+0.10	....	....
	−0.60	−0.50	−0.30	−0.30	....	....	−0.60	−0.40	−0.70	−0.20	....	....
Average deviation of shouldered from tapered	+0.10		−1.70				−1.20		−2.30			

TABLE I.—*Continued.*

	WG Wood Rosin		FF Wood Rosin		Helix Wood Rosin		WW Gum Rosin		M Gum Rosin		Synthetic Resin	
	Tapered Ring	Shouldered Ring	Tapered Ring	Shouldered Ring	Tapered Ring	Shouldered Ring	Tapered Ring	Shouldered Ring	Tapered Ring	Shouldered Ring	Tapered Ring	Shouldered Ring
	DR. R. J. SOBATZKI (PFLAUMER AND SCHREIBER) RESINOUS PRODUCTS AND CHEMICAL CO.											
Individual determinations	76.00	76.00	73.00	73.00	138.50	138.50	72.50	72.00	77.00	77.00	145.50	146.00
	76.00	76.00	73.50	74.00	138.50	139.00	72.00	72.50	76.50	77.00	145.50	145.50
	76.50	77.00	73.00	73.00	138.50	138.50	73.00	73.00	77.00	77.00	145.00	146.00
	76.50	76.00	73.00	73.00	138.50	138.50	73.00	73.50	77.00	77.00	145.50	145.50
	....	....	....	....	....	....	73.00	73.00	....	....	....	....
Average	76.30	76.30	73.10	73.30	138.50	138.60	72.70	72.80	76.90	77.00	145.40	145.80
Max. deviation	0.50	1.00	0.50	1.00	0.00	0.50	1.00	1.50	0.50	0.00	0.50	0.50
Max. deviation from average	+0.20	+0.70	+0.40	+0.70	+0.00	+0.40	+0.30	+0.70	+0.10	+0.00	+0.10	+0.20
	−0.30	−0.30	−0.10	−0.30	−0.00	−0.10	−0.70	−0.80	−0.40	−0.00	−0.40	−0.30
Average deviation of shouldered from tapered	0.00		+0.20		+0.10		+0.10		+0.10		+0.10	
	V. E. GROTLISCH, NAVAL STORES SECTION, AGRICULTURAL MARKETING ADMINISTRATION, U. S. DEPT. OF AGRICULTURE											
Individual determinations	75.75	76.00	72.50	72.00	140.50	139.50	71.50	71.00	75.50	75.00	141.50	141.50
	75.75	76.00	72.50	72.00	140.50	140.00	71.50	71.00	75.50	75.00	141.50	142.00
	75.50	76.00	72.50	72.00	140.00	140.00	71.50	72.00	75.00	75.00	143.00	141.50
	75.50	76.00	72.50	72.00	139.50	141.00	71.50	71.50	75.00	75.00	143.00	141.50
Average	75.60	76.00	72.50	72.00	140.10	140.10	71.50	71.40	75.30	75.00	142.30	141.60
Max. deviation	0.25	0.00	0.00	0.00	1.00	1.50	0.00	1.00	0.50	0.00	1.50	0.50
Max. deviation from average	+0.15	+0.00	+0.00	+0.00	+0.40	+0.90	+0.00	+0.60	+0.20	+0.00	+0.70	+0.40
	−0.10	−0.00	−0.00	−0.00	−0.60	−0.60	−0.00	−0.40	−0.30	−0.00	−0.80	−0.10
Average deviation of shouldered from tapered	+0.40		−0.50		0.00		−0.10		−0.30		−0.70	
	I. E. KNAPP, CROSBY NAVAL STORES, INC.											
Individual determinations	75.20	75.20	72.90	73.00	139.50	140.10	73.60	73.50	77.20	77.40	135.80	137.20
	75.30	75.20	72.90	73.00	140.20	140.20	74.00	74.00	77.30	77.50	136.10	137.50
	75.10	75.10	72.90	73.00	140.00	140.00	73.60	73.60	77.00	77.20	138.30	139.00
	75.10	75.20	73.10	73.10	140.10	140.80	73.70	73.80	77.20	77.30	138.50	138.80
Average	75.20	75.20	73.00	73.00	140.00	140.30	73.70	73.70	77.20	77.40	137.20	138.10
Max. deviation	0.20	0.10	0.20	0.10	0.70	0.80	0.40	0.50	0.30	0.30	2.70	1.80
Max. deviation from average	+0.10	+0.00	+0.10	+0.10	+0.20	+0.50	+0.30	+0.30	+0.10	+0.10	+1.30	+0.90
	−0.10	−0.10	−0.10	−0.00	−0.50	−0.30	−0.10	−0.20	−0.20	−0.20	−1.40	−0.90
Average deviation of shouldered from tapered	0.00		0.00		+0.30		0.00		+0.20		+0.90	
	W. A. KIRKLIN, HERCULES POWDER CO.											
Individual determinations	74.70	74.80	73.20	74.00	135.50	138.50	72.70	73.00	78.00	78.00	143.20	144.40
	74.70	75.20	73.60	74.40	135.80	138.80	73.00	73.50	78.50	78.80	141.50	145.00
	74.50	75.40	73.70	74.20	138.00	138.00	75.00	75.30	79.00	79.50	143.50	141.90
	74.80	75.90	74.00	74.90	138.90	139.00	75.50	75.50	79.50	80.10	144.00	143.10
Average	74.70	75.30	73.60	74.40	137.10	138.60	74.10	74.30	78.80	79.10	143.00	143.60
Max. deviation	0.30	1.10	0.80	0.90	3.50	1.00	2.80	2.50	1.50	2.10	2.50	3.10
Max. deviation from average	+0.10	+0.60	+0.40	+0.50	+1.90	+0.40	+1.40	+1.20	+0.70	+1.00	+1.00	+1.40
	−0.20	−0.30	−0.40	−0.40	−1.60	−0.60	−1.40	−1.30	−0.80	−1.10	−1.50	−1.70
Average deviation of shouldered from tapered	+0.60		+0.80		+1.50		+0.30		+0.30		+0.60	
	SUMMARY OF INDIVIDUAL LABORATORY DETERMINATIONS											
Max. deviation obtained by any one laboratory	1.20	1.20	0.80	1.00	3.50	1.50	2.80	2.50	1.50	2.10	2.70	3.10
Max. deviation from average obtained by any one laboratory	+0.60	+0.70	+0.40	+0.70	+1.90	+0.90	+1.40	+1.20	+0.70	+1.00	+1.30	+1.40
	−0.60	−0.50	−0.40	−0.30	−1.60	−0.60	−1.40	−1.30	−0.80	−1.10	−1.40	−1.70
Max. deviation of shouldered from tapered obtained by any one laboratory	+0.60		+0.80		+1.50		+0.90		+0.90		+0.90	
	−0.20		−1.70		−0.00		−1.20		−2.30		−0.70	

TABLE I.—*Concluded.*

	WG Wood Rosin		FF Wood Rosin		Helix Wood Rosin		WW Gum Rosin		M Gum Rosin		Synthetic Resin	
	Tapered Ring	Shouldered Ring	Tapered Ring	Shouldered Ring	Tapered Ring	Shouldered Ring	Tapered Ring	Shouldered Ring	Tapered Ring	Shouldered Ring	Tapered Ring	Shouldered Ring
	SUMMARY OF AVERAGE FIGURES, ALL LABORATORIES											
Average of all individual laboratory averages	74.83	75.07	72.60	72.55	137.96	138.59	72.29	72.38	76.70	76.63	142.81	143.15
Max. deviation from average of all individual laboratory averages	+1.83 −1.47	+0.97 −2.06	+1.00 −1.10	+1.85 −1.85	+2.14 −1.86	+1.71 −2.09	+1.81 −1.79	+1.91 −1.59	+2.10 −2.00	+2.47 −1.63	+2.89 −5.61	+2.65 −5.05
Average deviation of shouldered from tapered ring by all laboratories	+0.24		−0.05		+0.63		+0.09		−0.07		+0.34	

REPORT OF SUBCOMMITTEE IV ON ACID AND SAPONIFICATION NUMBERS AND UNSAPONIFIABLE MATTER

The current year has not seen a great deal of progress in this subcommittee but it has not been entirely inactive. In the past, at various times, samples of rosin have been sent to members for collaborative tests. This necessarily takes a considerable amount of work in preparing samples and compiling results and also disturbs the normal operations in laboratories of the committee members. For that reason the chairman has had some special investigative work done in the Chicago research laboratories of Swift and Co. in order that something more definite may be offered to the committee before asking for collaborative work.

A year or so ago it was proposed to use electrometric titration for determining the saponification number of dark products that screened the color when using an internal indicator. It was finally determined, however, that it would be necessary to give up the idea of electrometric titration because, for reasons not definitely determined, the electrometric titration did not give uniform results when compared with the titration from the internal indicator on different grades of rosin of sufficiently light color to permit the use of an internal indicator.

While a great amount of work was done trying to develop a method whereby the electrometric titration would be reliable, it was not found possible to devise any technique that would produce reliable results. Some results obtained by electrometric titration are shown in Table I, not as a recommendation for the method but to make the information available.

TABLE I.—RESULTS OF COLLABORATIVE TESTS ON SAPONIFICATION NUMBER OF ROSIN.

Rosins Analyzed		Results of Swift and Co.			Result of Naval Stores Research Division	
		Internal Indicator	External Indicator	Electrometric Titration	Internal Indicator	External Indicator
WW	Slash pine rosin.	171	175	177	175	177
WW	Slash pine rosin containing some aluminum rosinate...........	174	173	178	177	....
M	Slash pine rosin containing some iron rosinate....	173	174	184	174	....
D	Slash pine rosin containing more iron rosinate....	173	176	177	177	....
B	Oxidized slash pine rosin......	204	204	242	217	211 to 216
X	Wood rosin.....	171	173	190	174	175
K	Wood rosin.....	181	183	185	179	180
B	Wood rosin.....	161	161	177	...	153 to 155
	Vinsol..........	173[a]	173	202	173[a]	172 to 177

[a] Color of solution made determination of end point difficult. Operator was not sure of end point.

Previously, external indicators were tried without a great deal of success but now a technique has been found that will yield reliable results when using an external indicator. While the technique is not new it is not as common as the use of a spot plate. The technique amounts simply to saturating a filter paper with standard phenolphthalein indicator, then during the titration as the end point is approached a streak of the solution is placed on the sensitized filter paper by means of the stirring rod. Table I shows the results obtained by two collaborating laboratories using the external indicator as indicated above along with internal indicator and electrometric titration.

A further examination of the data presented in Table I shows favorable agreement between the results from the two collaborating laboratories on most determinations with the exception of the electrometric titration. It should be stated, however, that even though the internal indicator in most cases checked the external indicator there was nothing certain about the end point. There was considerable uncertainty in detecting the end point with the internal indicator, but there was no difficulty in detecting the end point with the external indicator. The external indicator is not recommended for light-colored rosins but it is recommended for the dark-colored rosins.

With these two collaborating laboratories showing checking results on dark rosin by the use of the external indicator, there are now in preparation four samples of rosin varying from a light to a dark color including a sample of Vinsol resin, and it is proposed to send these samples to all members of the commitee for determination of both the saponification and acid numbers. It is not expected that these results will be available for the annual meeting but they will be reported as soon as the tests are completed and the data are tabulated.

The subcommittee has not attempted to make any progress on the determination of unsaponifiable matter in rosins, since there is some question as to whether this determination is essential or necessary. It is possible that some of the members of the committee may know of very excellent reasons for having an unsaponifiable method and if so, the subcommittee would appreciate having some information on the importance of the unsaponifiable determination in rosin.

Respectfully submitted on behalf of the subcommittee,

C. C. ZEIGLER,
Chairman.

REPORT OF COMMITTEE D-18
ON
SOILS FOR ENGINEERING PURPOSES

Committee D-18 on Soils for Engineering Purposes has held two meetings during the past year: in Chicago, Ill., on June 27, 1941, and in Baltimore, Md., on December 2, 1941. The meeting scheduled for March 3, 1942, in Cleveland was cancelled due to other activities of the membership, as the result of the National Emergency.

As stated in the 1941 report of the committee, plans were made for a Symposium on Tests of Soils for Engineering Purposes to be presented at the 1942 annual meeting of the Society. These plans were considered at the December 2 meeting. The consensus of the members present was that due to the war effort and to the fact that more study was deemed necessary on some of the methods being proposed, this symposium should be placed in the hands of a special committee under the chairmanship of F. C. Lang to handle the details.

The Proposed Definitions of Terms and Symbols Relating to Soil Mechanics[1] which were published as information as an appendix to the 1941 report of Committee D-18 were reviewed by the committee during the year and a few editorial changes made. Committee D-18 recently presented these definitions, as editorially revised, to the Society through Committee E-10 on Standards for publication as tentative. This recommendation was accepted[2] by Committee E-10 on May 20, 1942, and the new tentative definitions have been published in separate pamphlet form under the designation D 653 – 42 T and will appear in the 1942 Book of A.S.T.M. Standards, Part II.

Subcommittee IV on Methods of Testing the Physical Characteristics of Soils (Paul Rapp, chairman) submitted to the committee a proposed Method of Test for Moisture-Density Relations of Soils.[3] This proposed method has been submitted to letter ballot of the committee which consists of 78 members; 63 members returned their ballots, of whom 53 have voted affirmatively, 6 negatively, and 4 members marked their ballots "not voting."

However, at the last annual meeting, this method was submitted to Committee D-4 on Road and Paving Materials for concurring action. Committee D-4 has not as yet acted on this method so that it is not ready for presentation at the annual meeting.[3]

Several subcommittees are studying tests with the expectation that reports on them will be ready for submittal to the Society at the 1942 annual meeting.

The committee has under consideration a proposed addition to Section 14 of the Standard Method of Mechanical Analysis of Soils (D 422 – 39) and a revision of the Tentative Methods of Surveying and Sampling Soils for

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 460 (1941).

[2] In submitting this recommendation to Committee E-10 on Standards, Committee D-18 reported the results of the letter ballot vote as follows: Of a voting membership of 78, 46 members returned their ballots, of whom 38 voted affirmatively, 0 negatively, and 8 members marked their ballots "not voting."

[3] See Editorial Note, p. 450.

Highway Subgrades (D 420 – 40 T). These recommendations will be presented to the Society later in the year through Committee E-10 on Standards.[3]

No modifications with respect to the Tentative Methods of Test for Moisture-Density Relations of Soil-Cement Mixtures (D 558 – 40 T), for Wetting-and-Drying Test of Compacted Soil-Cement Mixtures (D 559 – 40 T) or for Freezing-and-Thawing Test of Compacted Soil-Cement Mixtures (D 560 – 40 T) are contemplated at this time. The committee accordingly recommends that these three methods be continued as tentative without revision.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, C. A. Hogentogler.
Vice-Chairman, E. J. Kilcawley.
Secretary, Wesley R. Nelson.

This report has been submitted to letter ballot of the committee which consists of 78 members; 53 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

C. A. HOGENTOGLER,
Chairman.

WESLEY R. NELSON,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee D-18 presented to the Society through Committee E-10 on Standards the following recommendations:

Revision of Tentative Methods of:
Surveying and Sampling Soils for Highway Subgrades (D 420 – 40 T), and

Tentative Revision of Standard Method of:
Mechanical Analysis of Soils (D 422 – 39).

These recommendations were accepted by Committee E-10 on August 24, 1942, and the revised methods and the tentative revision appear in the 1942 Book of A.S.T.M. Standards, Part II, pp. 1260 and 1444.

The proposed Method of Test for Moisture-Density Relations of Soils, submitted jointly by Committees D-4 and D-18, were accepted by Committee E-10 on September 17, 1942, for publication as tentative. This new tentative method was assigned the designation D 698 – 42 T and appears in the 1942 Book of A.S.T.M. Standards, Part II, p. 1280.

REPORT OF COMMITTEE D-19

ON

WATER FOR INDUSTRIAL USES

Committee D-19 on Water for Industrial Uses and its subcommittees held meetings at Chicago, Ill., on June 25 and 26, 1941. No additional meetings were held by any of the subcommittees during the year.

The committee sponsored a session on water at the 1941 annual meeting in the form of a Symposium on Problems and Practice in Determining Steam Purity by Conductivity Methods, in which six papers[1] were presented and discussed.

The committee is sponsoring a water session at this annual meeting of the Society, in the form of a Round-Table Discussion on The Solvent Action of Water Vapor at High Temperatures and Pressures. The topic will be introduced by George W. Morey, Geophysical Laboratory, Carnegie Institute of Washington, Washington, D. C.

The Society was represented by Max Hecht at a meeting held December 3, 1941, of the Subcommittee on Care of Steam Boilers in Service, of the Boiler Code Committee of the American Society of Mechanical Engineers. Plans were furthered by the organized section for the revision of the paragraphs on water chemistry that now appear in the A.S.M.E. Boiler Construction Code, 1940 Edition, especially in Section VII, on Suggested Rules for Care of Power Boilers.

The Society was represented by Max Hecht and S. F. Whirl, the latter representing F. N. Speller, at the meeting of the Executive Committee of the Joint Research Committee on Boiler Feed Water Studies, held on December 3, 1941, in New York, N. Y.

Committee D-19 has recommended to the Society that the A.S.T.M. Standard Method of Reporting Results of Analysis of Industrial Waters (D 596 - 41) be referred to the American Standards Association for approval as American standard. The officers of the committee have received a number of favorable comments concerning the use of this improved method of reporting from several of the national societies and other organizations.

ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the following tentative methods be approved, without change, for reference to letter ballot of the Society for adoption as standard:

Tentative Methods for:[2]

Determination of the Calcium Ion and Magnesium Ion in Industrial Waters (D 511 - 40 T),
Determination of the Chloride Ion in Industrial Waters (D 512 - 40 T), and
Determination of the Sulfate Ion in Industrial Waters (D 516 - 40 T).

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 65 members; 53 members returned their ballots, of whom 51 have voted affirmatively, 0 negatively, and 2 mem' bers marked their ballots "not voting.'

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 41, pp. 1261, 1264, 1277, 1285, 1302, 1314, and 1326 (1941).

[2] 1940 Supplement to Book of A.S.T.M. Standards, Part III, pp. 529, 533, 539.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II, Editorial (E. P. Partridge, chairman).—This subcommittee did not meet during the year but the chairman assisted in the review of the papers presented at the 1941 water session.

Subcommittee III on Methods of Sampling (D. K. French, chairman).—A meeting of this subcommittee was held on June 25, 1941, in Chicago, Ill. The subcommittee chairman has studied the replies received from the members concerning their individual methods for collection of samples of water and condensed steam from boilers and other equipment. These replies show a wide diversity of individual methods. Nevertheless, new inquiries have been circulated, and from the replies, it is intended to formulate methods of sampling of water and condensed steam from equipment. These replies will be reviewed at the 1942 meeting of the subcommittee.

Subcommittee IV on Methods of Analysis (H. C. Miller, chairman).—A meeting of this subcommittee was held on June 25, 1941, in Chicago, Ill. The chairman has prepared drafts of proposed methods of analysis of certain substances in water, and the form and substance will be discussed at the 1942 meeting of the subcommittee. Should the content of the various methods be favorably accepted, the subcommittee proposes to have the material published as information.

Subcommittee V on Classification (R. C. Adams, chairman).—A meeting of this subcommittee was held on June 25, 1941, in Chicago, Ill. The preliminary plans for the classification of water intended for industrial uses, will be discussed at the meeting of the subcommittee during this annual meeting. A limited survey of this work has been under way, and it is expected as a result of this study, that the subcommittee will be able to further its plans.

The present officers of the committee were re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee, which consists of 65 members; 45 members returned their ballots, of whom 44 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

MAX HECHT,
Chairman.

R. E. HALL,
Secretary.

REPORT OF COMMITTEE D-20

ON

PLASTICS

Since the June, 1941, meeting in Chicago, Ill., Committee D-20 on Plastics and its subcommittees have held two meetings: one in New York, N. Y., in October, 1941, and one in Philadelphia, Pa., in February, 1942. The committee expects to hold a further meeting in Atlantic City, N. J., during the 1942 annual meeting.

The scope of Committee D-20 has been broadened to include a Subcommittee VI on Specifications. G. M. Kline resigned as chairman of Subcommittee V on Permanence Properties and was appointed chairman of the new Subcommittee VI on Specifications; J. H. Teeple was appointed chairman of Subcommittee V.

Committee D-20 now has 75 voting members, representing an increase during the year of 3 voting members.

Subsequent to the 1941 annual meeting, Committee D-20 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative Methods of:

Test for Flammability of Plastics Over 0.050 in. in Thickness (D 635 – 41 T),
Test for Diffusion of Light by Plastics (D 636 – 41 T),
Test for Surface Irregularities of Flat Transparent Plastic Sheet (D 637 – 41 T),
Tension Testing of Plastics (D 638 – 41 T),

Revision of Tentative Method of:

Test for Resistance of Plastics to Chemical Reagents (D 543 – 39 T), and

Tentative Revision of Standard Method of:

Test for Impact Resistance of Electrical Insulating Materials (D 256 – 38) in the form of new Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (D 256 – 41 T), submitted jointly with Committee D-9 on Electrical Insulating Materials.

These recommendations were accepted[1] by Committee E-10 on August 25, 1941, and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part III.

I. NEW TENTATIVE STANDARDS

The committee recommends that the following new proposed methods of test and descriptive nomenclature be accepted for publication as tentative:[2]

Test for Repeated Flexural Stress (Fatigue) of Plastics,
Test for Mar Resistance of Plastics,
Recommended Practice for Long-Time Tension Tests of Plastics,
Test for Haze of Transparent Plastics by Photoelectric Cell, and
Classification of Terms and Descriptive Nomenclature of Objects Made from Plastics.

II. ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the following methods of test be approved for reference to letter ballot of the Society for adoption as standard:

Tentative Methods of Test for:

Index of Refraction of Transparent Organic Plastics (D 542 – 40 T),[3] and
Water Absorption of Plastics (D 570 – 40 T).[4]

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee D-20 reported the following results of the letter ballot vote of 45 and 52 ballots returned respectively from a committee membership of 70: D 635, affirmative 38, negative 2, ballots marked "not voting" 5; D 636, affirmative 29, negative 0, ballots marked "not voting" 16; D 637, affirmative 32, negative 0, ballots marked "not voting" 13; D 638, affirmative 39, negative 5, ballots marked "not voting" 8; D 543, affirmative 46, negative 0, ballots marked "not voting" 6; D 256, affirmative 44, negative 2, ballots marked "not voting" 6.

[2] These methods and classification were accepted as tentative by the Society and appear in the 1942 Book of A.S.T.M. Standards, Part III, pp. 1251, 1263, 1284, 1260, and 1288, respectively.

[3] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 289.

[4] *Ibid.*, p. 292.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 77 voting members, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Test for Repeated Flexural Stress (Fatigue) of Plastics	40	1	11
Test for Mar Resistance of Plastics	40	1	9
Long-Time Tension Tests of Plastics	36	3	13
Test for Haze of Transparent Plastics by Photoelectric Cell[a]	36	4	12
Classification of Terms and Descriptive Nomenclature of Objects Made from Plastics	47	3	2
II. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Test for Index of Refraction of Transparent Organic Plastics (D 542 – 40 T)	32	0	18
Test for Water Absorption of Plastics (D 570 – 40 T)	43	0	7

[a] The classified vote on the Proposed Tentative Method of Test for Haze Transparent Plastics by Photoelectric Cell was as follows: Affirmative: 13 producers, 8 consumers, 15 general interests; Negative: 3 producers, 1 consumer, 0 general interests.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Strength Properties (M. H. Bigelow, chairman).—Through the efforts of this subcommittee, the following methods of test are now available: Tentative Method of Tension Testing of Plastics (D 638 – 41 T) and Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (D 256 – 41 T). This subcommittee prepared the proposed Tentative Method of Test for Repeated Flexural Stress (Fatigue) of Plastics, referred to elsewhere in this report. It has under study tests for determining modulus of elasticity, shear, compressive strength,[5] flexural strength, bearing strength, repeated and high-speed impact strength, and the simplification of the tension test for plastics.

Subcommittee II on Hardness Properties (W. H. Gardner, chairman).—This subcommittee prepared the Tentative Method of Test for Deformation of Plastics Under Load at Elevated Temperatures (D 621 – 41 T) and has developed the Method of Test for Mar Resistance of Plastics as well as the Recommended Practice for Long-Time Tension Tests of Plastics, being submitted as tentative as referred to earlier in this report. Work is now under way on the simplification of a heat distortion test and a study is being made of scratch resistance, wear resistance of plastic bearings, and machinability of plastics.

Subcommittee III on Thermal Properties (W. A. Zinzow, chairman).—In accordance with the recommendations of this subcommittee, the Tentative Methods of Test for Measuring Flow Temperatures of Thermoplastic Molding Materials and for Flammability of Plastics 0.050 in. and Under in Thickness have been revised and now bear the designations D 569 – 41 T and D 568 – 41 T, respectively. Round robin tests are now being conducted on thermosetting plastics and a study is being made of the changes in plastics on heating as well as thermal expansion.

Subcommittee IV on Optical Properties (W. F. Bartoe, chairman).—This subcommittee is responsible for the preparation of the Tentative Methods of Test for Diffusion of Light by Plastics (D 636 – 41 T) and for Surface Irregularities of Flat Transparent Plastic Sheet (D 637 – 41 T). The proposed Tentative Method of Test for Haze of Transparent Plastics by Photoelectric Cell and the Classification of Terms and Descriptive Nomenclature of Objects Made from Plastics, mentioned earlier in this report, were prepared by this subcommittee. Studies are being made of the optical clarity and associated characteristics of plastics.

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[5] See Editorial Note, p. 455.

Subcommittee V on Permanence Properties (J. H. Teeple, chairman).—The Tentative Method of Test for Colorfastness of Plastics to Light (D 620 – 41 T) was developed by this subcommittee and its Tentative Method of Test for Water Absorption of Plastics (D 570 – 40 T) is now being recommended for adoption as standard. The subcommittee is engaged in work leading to an accelerated service weathering test for plastics and is actively studying the effect of heat, water, and chemicals on plastics.

Subcommittee VI on Specifications (G. M. Kline, chairman).—This new subcommittee has been quite active in collaboration with the various Government agencies requiring specifications. It has mostly rendered service to the Quartermaster Corps, Ordnance Department of the War Department, Navy Bureau of Aeronautics, and the Procurement Division of the Treasury Department. The subcommittee expects to formulate specifications for the various thermoplastics as well as thermosetting materials. It is cooperating with Subcommittee II, of Committee D-9, on specifications for phenolic molding materials.

At the February meeting in Philadelphia, the following officers were unanimously elected for the ensuing term of two years:

Chairman, Robert Burns.
First Vice-Chairman, H. K. Nason.
Second Vice-Chairman, Donald Gibb.
Secretary, L. W. A. Meyer.

This report has been submitted to letter ballot of the committee which consists of 75 members; 50 members returned their ballots, of whom 36 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. E. EMLEY,
Chairman.

W. A. EVANS,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee D-20 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative Methods of Test for:
Compressive Strength of Plastics (D 695 – 42 T),
Coefficient of Linear Thermal Expansion of Plastics (D 696 – 42 T),
Water Vapor Permeability of Plastic Sheets (D 697 – 42 T), and

Revision of Tentative Methods of:
Tension Testing of Plastics (D 638 – 41 T).

These recommendations were accepted by Committee E-10 on August 24, 1942, and the new and revised tentative methods appear in the 1942 Book of A.S.T.M. Standards, Part III, pp. 1268, 1276, 1280, and 1241.

REPORT OF COMMITTEE E-1
ON
METHODS OF TESTING

Committee E-1 on Methods of Testing held a meeting in Chicago, Ill., on June 26, 1941, during the annual meeting of the Society. A number of the technical committees and their sections have been active and furthered their work at meetings held last June and during the A.S.T.M. spring group committee meetings held in Cleveland, Ohio, the week of March 3, 1942. Additional meetings of the technical committees and sections and of Committee E-1 will be held prior to the presentation of this report to the Society.

The Executive Committee of the Society appointed three representatives-at-large to serve on the Advisory Committee, namely, W. H. Fulweiler, Consulting Chemist, H. F. Moore, University of Illinois, and T. S. Fuller, General Electric Co., each for a three-year term, expiring in 1944.

Subsequent to the 1941 annual meeting, Committee E-1 presented to the Society through Committee E-10 on Standards, the following recommendations, which were accepted[1] by Committee E-10 on August 25, 1941, and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part I:

Revision of Tentative:
Methods of Impact Testing of Metallic Materials (E 23 - 40 T), and
Specifications for A.S.T.M. Thermometers (E 1 - 40 T).
Tentative Revision of Standard:
Specifications for A.S.T.M. Thermometers (E 1 - 39).

EMERGENCY STANDARD

The Section on Indentation Hardness, of Technical Committee I, completed a Hardness Conversion Table for Cartridge Brass which gives relations between diamond pyramid hardness, Rockwell hardness, and Brinell hardness. This table was published as information in the March, 1942, issue of the ASTM BULLETIN. In view of the pressing need for these hardness conversion data on cartridge brass, the conversion table was submitted for publication as an emergency standard. This recommendation was accepted by Committee E-10 on April 10, 1942, and the Emergency Standard issued under the designation ES - 4.

I. NEW STANDARD

Proposed Standard Hardness Conversion Table for Cartridge Brass.—The Hardness Conversion Table for Cartridge Brass[2] issued in April as Emergency Standard ES - 4 is now recommended for formal adoption as standard and the necessary nine-tenths affirmative vote at the annual meeting is requested in order that this recommendation may be referred to Society letter ballot.

II. REVISION OF TENTATIVE STANDARD

Tentative Specifications for A.S.T.M. Thermometers (**E 1 - 41 T**).[3]—In the 1941 Report[4] there was published as information a reference to methods of determining the ice point in the standardization of kinematic viscosity thermometers. It is now recommended that reference to these methods be included as a note in the Tentative Specifications for A.S.T.M. Thermometers (E 1 - 41 T) to appear in connection with the requirements for the three kinematic viscosity thermometers E 1 (28 F - 39 T), E 1 (29 F - 39 T), and E 1 (30 F - 39 T).

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee E-1 reported the following results of the letter ballot vote of a total of 36 ballots returned from a committee membership of 52: E 23 - 40 T, affirmative 24, negative 0, ballots marked "not voting" 12; E 1 - 40 T, affirmative 17, negative 0, ballots marked "not voting" 19; E 1 - 39, affirmative 17, negative 0, ballots marked "not voting" 19.

[2] These tables were adopted as standard by the Society and appear in the 1942 Book of A.S.T.M. Standards, Part I, p. 925.

[3] 1941 Supplement to Book of A.S.T.M. Standards, Parts II and III.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 481 (1941).

III. Revision of Standard, Immediate Adoption

Standard Specifications for A.S.T.M. Thermometers (**E 1 - 39**).[5]—The following revisions are recommended for immediate adoption and the committee asks for a nine-tenths affirmative vote at the annual meeting in order that this recommendation may be referred to Society letter ballot. These revisions were published as information in the 1941 Report[4] and upon approval by the Society it is recommended that they become effective December 1, 1942.

High Distillation Thermometers.—In the A.S.T.M. High Distillation Thermometers E 1 (8C - 39) and E 1 (8F - 39) change the distance from the bottom of the bulb to the graduation line at 0 C. (32 F.) from the present specified dimension of "25 to 35 mm. (0.98 to 1.38 in.)" to a distance of "25 to 45 mm. (0.98 to 1.77 in.)."

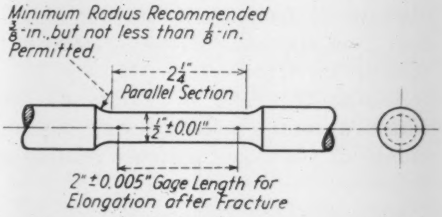
Open Flash Thermometers.—In the A.S.T.M. Open Flash Thermometers E 1 (11 C - 39) and E 1 (11 F - 39) reduce the length of the bulb from the present specified requirement of "not over 13 mm. (0.51 in.)" to read "not over 10 mm. (0.39 in.)."

IV. Adoption of Tentative Standards as Standard

The committee recommends that the following three tentative standards be approved for submission to letter ballot of the Society for adoption as standard without revision:

- Tentative Method of Test for Rockwell Superficial Hardness of Metallic Materials (E 18 - 39 T),
- Tentative Definitions of Terms Relating to Rheological Properties of Matter (E 24 - 40 T), and
- Tentative Specifications for A.S.T.M. Thermometers (E 1 - 41 T):
  - (*1*) Turpentine Distillation Thermometer E 1 (27C-40T), replacing Standard Thermometer E 1 (27C-39)
  - (*2*) Titer Test Thermometer E 1 (36C-41T).

The committee also recommends that the Tentative Methods of Tension Testing of Metallic Materials (E 8 - 40 T) be approved for submission to letter ballot of the Society for adoption as standard with the following revisions:


Minimum Radius Recommended $\frac{3}{8}$-in., but not less than $\frac{1}{8}$-in. Permitted.
$2\frac{1}{4}''$
Parallel Section
$\frac{1}{2}'' \pm 0.01''$
$2'' \pm 0.005''$ Gage Length for Elongation after Fracture

Note.—The gage length, parallel section, and fillets shall be as shown, but the ends may be of any shape to fit the holders of the testing machine in such a way that the load shall be axial.

Fig. 1.—Standard 2-in. Gage Length Tension Test Specimen.

Section 5.—Change to read as follows by the addition of the italicized figures and the omission of those in brackets:

> 5. The standard tension test specimen of rectangular cross-section with a 2-in. gage length is shown in Fig. 2. This specimen is used for testing ferrous and non-ferrous metals in the form of plate, sheet, flat wire, strip, band, and hoop ranging in nominal thickness from [0.01] *0.005* to, but not including, ½ in., and widths of $1\frac{1}{8}$ in. or greater.

Fig. 3.—Change the illustration of the standard round tension test specimen so that the radius at each end of the reduced section is ⅜ in., and so that it is clear that shouldered-end specimens should not be made by simply scaling the ends of the specimen as shown in the present Fig. 3. The revised illustration is shown in the accompanying Fig. 1.

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[5] 1939 Book of A.S.T.M. Standards, Part II, 838; Part III, p. 520.

Section 6 (b).—In order to clarify the first sentence change it to read as follows by the addition of the italicized words:

> It is desirable to have the diameter of the gage length *of the specimen shown in Fig. 3*, slightly smaller at the center than at the ends.

Transpose Paragraphs (*c*) and (*d*) of this section to appear as a new Section 8 (*a*) and (*b*) under the marginal heading "Round Tension Test Specimens," renumbering the remaining sections accordingly.

Fig. 6.—On the recommendation of Committee A-3 on Cast Iron, change the diameter of the machined section, dimension *c*, of specimen B from "0.800 in." to read "0.750 in."

Section 18.—Add to Paragraph (*a*) which covers the speed of testing requirements an additional sentence reading as follows:

> When the tensile properties of a material are appreciably affected by speed of straining suitable limits for rate of straining should be included in the purchase specifications covering that material.[a]

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[a] This is the responsibility of those preparing the purchase specifications.

Change Paragraph (*c*) to read as follows by the addition of the italicized word and the omission of those in brackets:

> (*c*) The change in tensile strength values produced by a change in the rate of straining is [usually considerably] *often* less than that observed for the yield strength. The range of permissible speeds is, therefore, wider.

Section 22 (a).—Change the fourth sentence to read as follows by the addition of the italicized words:

> The load at the "halt *in the gage*" or the "drop *of the beam*" is recorded and the corresponding stress is taken as the yield point.

Add a new Paragraph (*d*) to this section to read as follows:

> (*d*) *Autographic Diagram Method.*—When a sharp-kneed stress-strain diagram is obtained by an autographic recording device the stress corresponding to the top of the knee or the point at which the curve drops is to be taken as the yield point.

V. ADOPTION AS STANDARD OF TENTATIVE REVISION OF STANDARD

Standard Specifications for A.S.T.M. Thermometers (**E 1 - 39**).[5]—The tentative revision submitted in August, 1941,[6] of the A.S.T.M. Stability Test of Soluble Nitrocellulose Thermometer E 1 (26 C - 29) is recommended for adoption as standard and the committee accordingly asks for a nine-tenths affirmative vote at the annual meeting in order that this recommendation may be referred to Society letter ballot.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW STANDARD			
Hardness Conversion Table for Cartridge Brass	16	0	8
II. REVISION OF TENTATIVE STANDARD			
Spec. for A.S.T.M. Thermometers (E 1 - 41 T), kinematic viscosity thermometers	12	0	12
III. REVISION OF STANDARD, IMMEDIATE ADOPTION			
Spec. for A.S.T.M. Thermometers (E 1 - 39):			
1. High Distillation Thermometers E-1 (8C-39) and E-1 (8F-39)	11	0	13
2. Open Flash Thermometers E-1 (11C-39) and E-1 (11F-39)	11	0	13
IV. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Test for Rockwell Hardness of Metallic Materials (E 18 - 39 T)	16	0	8
Def. of Terms Relating to Rheological Properties of Matter (E 24 - 40 T)	17	0	7
Spec. for A.S.T.M. Thermometers (E 1 - 41 T):			
1. Turpentine Distillation Thermometer E-1 (27C-40T), replacing Standard Thermometer E-1 (27C-39)	11	0	13
2. Titer Test Thermometer E-1 (36C-41T)	11	0	13
Methods of Tension Testing of Metallic Materials (E 8 - 40 T), as revised	18	0	6
V. ADOPTION AS STANDARD OF TENTATIVE REVISION OF STANDARD			
Spec. for A.S.T.M. Thermometers (E 1 - 39), thermometer for stability test of soluble nitrocellulose	11	0	13

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[6] 1941 Supplement to Book of A.S.T.M. Standards, Part II, 426; Part III, p. 639.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 52 members; 24 have returned their ballots with the results shown in Table I.

ACTIVITIES OF TECHNICAL COMMITTEES

Technical Committee I on Mechanical Testing (H. F. Moore, chairman):

The activities of the various sections of this technical committee have been fewer this year than is customary. This is due very largely to the war situation and the heavy demands it has made upon the time of practically all the members of these sections. Furthermore, with the exception of needed emergency standards, it seemed a good policy to make as few changes in standards during the present emergency as possible.

Section on Tension Testing (R. L. Templin, chairman).—This section held a meeting at Cleveland, Ohio, on March 4, 1942, at which time the Tentative Methods of Tension Testing of Metallic Materials (E 8 – 40 T) were reviewed and it was voted to recommend that with the changes given earlier in this report these tentative methods be adopted as standard.

Favorable action on this recommendation has been taken by the Advisory Committee of Technical Committee I.

Section on Compression Testing (M. F. Sayre, chairman).—This section will meet at Atlantic City, N. J., to consider the revision of the Tentative Methods of Compression Testing of Metallic Materials (E 9 – 33 T) so that the requirements for testing bearing metal and the proposed compression test procedure for cast iron may be brought into harmony.

Section on Indentation Hardness (J. R. Townsend, chairman).—The Hardness Conversion Table for Cartridge Brass, issued in April as Emergency Standard ES – 4 and now recommended for formal adoption as standard as mentioned earlier in this report, was prepared by a special group of this section under the chairmanship of R. L. Kenyon. In the preparation of the conversion tables, the committee carried on a round-robin series of tests and made an extensive survey of existing hardness test data on this copper alloy. The conversion table has already proved very useful in the war effort and its appearance has been very timely.

Additional work on hardness conversion relationships of the hardened or heat-treated steels is now under way by another subgroup also under the chairmanship of Mr. Kenyon. The other members of this committee include W. E. Ingerson, V. E. Lysaght, L. H. Fry, R. H. Heyer, T. H. Wickenden representative of the Society of Automotive Engineers, and Howard Scott representative of the American Society for Metals. The subgroup is making very satisfactory progress on this project and may have a recommendation to present to the committee later in the year.

Studies carried on in connection with the work of this committee are described in a paper by R. H. Heyer on "Hardness Conversion Relationships"[7] being presented at this annual meeting.

The section has approved by letter ballot the adoption as standard without revision of the Tentative Method of Test for Rockwell Superficial Hardness of Metallic Materials (E 18 – 39 T). The Standard Methods of Test for Rockwell Hardness of Metallic Materials (E 18 – 36) and for Brinell Hardness of Metallic Materials (E 10 – 27) have also been reviewed and it is recommended that they be continued without change.

Section on Testing of Thin Sheet Metals (C. H. Marshall, chairman).—This section has asked to be discharged, and this request has been approved by the Technical Committee on Mechanical Testing and presented to Committee E-1. In discharging this section, it is desired to record the appreciation of the Committee on Mechanical Testing and the main committee for the excellent work done by this section in designing the most carefully thought-out tension specimen found in the A.S.T.M. standards. Mr. C. H. Marshall, chairman of the section, will continue as a member of the Advisory Committee of the Technical Committee on Mechanical Testing.

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[7] See p. 708.

Section on Calibration of Testing Machines and Apparatus (H. F. Moore, chairman).—This section has before it a proposed tentative standard for calibration and classification of strainometers. Inasmuch as there are very few laboratories equipped to calibrate strainometers accurately, and as the introduction of such a tentative standard might require a considerable amount of apparatus building and some additional load on inspectors of materials, it is felt that during the present war situation, or at least for the present year, it may be well to defer the completion of this as a tentative method. It was decided, however, to submit it for publication as information in the form of a proposed method.[8]

The section has reviewed the Standard Methods of Verification of Testing Machines (E 4 – 36) and has no changes to suggest in it at this time.

Technical Committee II on Consistency, Plasticity, and Related Properties (Eugene C. Bingham, chairman):

The studies being carried out at the Brooklyn Polytechnic Institute on the ring-and-ball softening point test and its possible correlation with absolute methods has been carried on in accordance with a program arranged by the Special Research Committee on Industrial Flow Tests under the chairmanship of Robert Burns. It was hoped that the tests might have been completed to permit presentation of a paper at this annual meeting. Due to the demands on the time of the investigators as a result of the war effort, this was not found possible, and a report on this investigation will accordingly not be available until 1943.

The Tentative Method of Test for Softening Point (Ball and Tapered Ring Apparatus) (E 28 – 39 T) is to be revised[9] as the result of further studies on the use of a shouldered ring proposed by Committee D-17 on Naval Stores. A series of tests on samples of rosin using the shouldered ring is presented this year in the current report of Committee D-17.[10]

The Tentative Definitions of Terms Relating to Rheological Properties of Matter (E 24 – 40 T) have been approved by letter ballot of this subcommittee for adoption as standard.

Technical Committee III on Particle Size and Shape (L. T. Work, chairman):

The Section on Pigment-Type Materials (C. E. Barnett, chairman) is continuing its studies of methods of measuring particle size by microscopic methods and until this work is completed the Tentative Method of Test for the Particle Size Distribution of Subsieve Size Particulate Substances (E 20 – 33 T) is being continued in its present status.

The Section on Sieves (A. A. Klein, chairman) reports that the revisions made this year in the Federal Specification for Standard Testing Sieves (RR-S-366) bring this specification into agreement with the A.S.T.M. Standard Specifications for Sieves for Testing Purposes (E 11 – 39).

Technical Committee IX on Interpretation and Presentation of Data (H. F. Dodge, chairman):

This technical committee has under consideration two additions to the A.S.T.M. Manual on Presentation of Data.

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[8] Proposed Method of Verification and Classification of Strainometers, ASTM BULLETIN, No. 117, August, 1942, p. 83.

[9] See Editorial Note, p. 462.

[10] See p. 443.

These deal, respectively, with the sampling errors of averages as a third supplement to the manual, and a fourth supplement covering the computation of correlation coefficients. Two charts submitted by the manual committee which cover three-sigma control limits for "number of defects" and for "fraction defective" are also being considered for inclusion in a future revision of the manual.

Technical Committee X on Conditioning and Weathering (Robert Burns, chairman):

In addition to maintaining close contact with all standing committees concerned with conditioning and weathering, this committee has made substantial progress toward the standardization of several items of importance in this field.

The committee has prepared a proposed Tentative Recommended Practice for Characteristics of Standard Carbon Arc Accelerated Weathering Unit[9] which sets forth the basic features desirable in apparatus of this type. While it is recognized that the establishment of precise weathering cycles covering specific materials for specific locations is the function of the standing committees, it is felt that this standard will establish certain fundamental principles which any carbon arc weathering device should possess.

The committee has also completed proposed Tentative Definitions with Procedures Relating to Conditioning and Weathering.[9] This is a compilation of items adopted by various standing committees and now proposed for general acceptance principally on the basis of wide applicability, and after careful review by this technical committee.

Technical Committee XI on Designation and Interpretation of Numerical Requirements (J. A. Gann, chairman):

The Tentative Recommended Practices for Designation of Numerical Requirements in Standards (E 29 – 40 T) developed by this technical committee are being continued as tentative pending completion of considerations now being given to further clarifying the use of the expressions "plus *or* minus" *versus* "plus *and* minus" in connection with tolerances or permissible variations prescribed in A.S.T.M. standards. The need for a general statement or recommendation regarding reasonable deviations when the required precision does not call for or require an exact statement of necessary tolerances is also being studied.

Technical Committee XII on Laboratory Apparatus (W. H. Fulweiler, chairman):

The work of this technical committee has been carried on by its six sections although the demands of the war effort have affected, to some extent, the amount of time which the members have been able to devote to committee work. The scarcity of materials used in the manufacture of apparatus has also presented the committee with a number of problems.

Section A on Thermometers (R. M. Wilhelm, chairman).—This section held a meeting in Cleveland during the A.S.T.M. spring group committee meetings at which time action was taken on a number of recommendations affecting the Specifications for A.S.T.M. Thermometers (E 1 – 39 and E 1 – 41 T). These various recommendations are referred to earlier in this report.

Section B on Hydrometers (J. P. Bader, chairman).—The third in a series of papers sponsored by Technical Committee XII has been prepared by J. P. Bader and E. L. Peffer on "Methods of Testing Hydrometers" for publication as an Appendix to this report.

Section C on Volumetric Glassware (J. J. Moran, chairman) and *Section D on Laboratory Glassware* (F. F. Shetterly, chairman).—These sections have continued to cooperate with the standing committees in the review of new and revised methods containing requirements for glass apparatus.

Section F on Metalware Apparatus (J. E. Patterson, chairman).—Members of this section have reviewed during the year the requirements for certain metalware apparatus, particularly as concerns the use of stainless steel which is now very difficult to obtain and in some instances is no longer available for apparatus manufacture. These matters are being discussed with the originating standing committee and in one case it is expected that an emergency alternate provision will be issued providing for the use of glass in place of stainless steel.

Technical Committee XIII on Hydrogen Ion Determinations (E. B. Ashcraft, chairman):

This technical committee was organized in June, 1941, and during the past year it has reviewed certain tests for pH value prepared by other Society committees. The principal function of this committee will be to serve in an advisory capacity to the standing committees of the Society on problems dealing with pH measurement and as a clearing house for information on hydrogen ion tests.

The present chairman and vice-chairman were re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 52 members; 25 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. H. FULWEILER,
Chairman.

P. J. SMITH,
Ex-officio Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee E-1 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative:
- Definitions with Procedures Relating to Conditioning and Weathering (E 41 - 42 T),
- Recommended Practice for Characteristics of Standard Carbon Arc Accelerated Weathering Unit (E 42 - 42 T), and

Revision of Tentative Method of Test for:
- Softening Point (Ball and Tapered Ring Apparatus) (E 28 - 39 T).

These recommendations were accepted by Committee E-10 on August 24, 1942, and the new and revised tentative standards appear in the 1942 Book of A.S.T.M. Standards, Part II, pp. 1434, 1432, 1419; Part III, pp. 1581, 1579, and 1566.

APPENDIX

METHODS OF TESTING HYDROMETERS

By J. P. Bader[1] and E. L. Peffer[2]

Part I—Prepared by J. P. Bader

The determination of specific gravity of liquids can be made by the following three general methods:

Method 1.—By weighing at standard temperature in glass containers of known volume (pycnometers).

Specific gravity is defined as the ratio of the weight of a certain volume of the liquid in question to the weight of the same volume of water taken as standard at a stated temperature.

Pycnometers of the usual design are somewhat conical in shape and fitted with a ground-glass stopper with a hole in the stopper or fitted with a thermometer serving as a stopper and a side tube for permitting the removal of the excess liquid. Care must be taken, however, to set the stopper uniformly to avoid the possibility of error due to variations in the volume of the pycnometer. Others of special design for specific purposes have been found to be very useful and may be designed to meet special requirements.

The buoyancy correction to be applied to any apparent weight, to reduce it to true weight in vacuum, is calculated by multiplying the air density by the difference in volumes between the object weighed and the weights required to balance it on an even-arm balance; or

$$B = \rho(V - V_W)$$

where

B = air buoyancy,
ρ = air density,
V = volume of object weighed, and
V_w = volume of weights.[3]

Method 2.—By use of hydrostatic balances, that is, weighing or balancing a plummet of known weight and displacement in the liquid under test. The Westphal Balance is the most generally known device of this kind.

The use of this method is limited to liquids of such viscosity, that the free movement of the sinker in the liquid is not appreciably retarded. In the case of the Westphal Balance and other special balances of this type the sinker or plummet is usually of small volume so that the accuracy is limited. Their principal advantages are that samples of small volume may be used for specific gravity determinations.

Method 3.—Instruments of flotation—hydrometers.

The purpose of this paper is to describe a convenient means of testing hydrometers in the laboratory. The basic principle of the hydrometer is believed to have been discovered in the third century B.C. by Archimedes (287 to 212 B.C.), and a crude form of this device is attributed to him. No record of development of this instrument appears until the latter part of the Seventeenth Century, when it was invented by Robert Boyle. Various forms of the hydrometer have been made out of ivory, metal, and glass, some of which are illustrated below, but for the purpose of this paper only the conventional, glass, constant-mass, variable-displacement type (Figs. 4 to 9, inclusive), which has the most common use in the laboratory, will be considered here.

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[1] President, The Emil Greiner Co., New York, N. Y.

[2] Chief, Capacity and Density Section, National Bureau of Standards, Washington, D. C.

[3] For detailed discussion see paper by H. W. Bearce, "Standardization of Methods for the Determination of Density," *Proceedings*, Am. Soc. Testing Mats., Vol. XIX, Part I, p. 412 (1919).

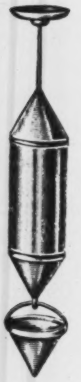

FIG. 1

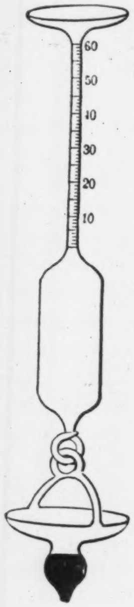

FIG. 2

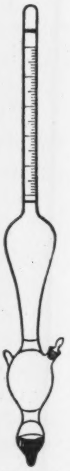

FIG. 3

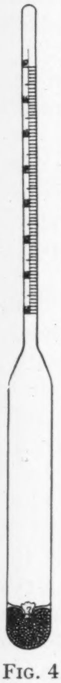

FIG. 4

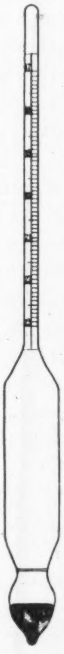

FIG. 5

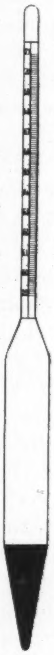

FIG. 6

Figures 1 and 2 illustrate the so-called Nicholson hydrometer, a now obsolete type used for determining specific gravity of solid particles in which weighed particles are placed in the basket after which a reading is taken in water or other sustaining fluid, using weights on the platform and position of submersion of the scale as a means of evaluating the specific gravity of the solid. These were made either of metal or glass.

Figure 3 illustrates an obsolete type used in determining the specific gravity of liquids where the amount available was too small to float an hydrometer. The liquid was enclosed in the chamber just above the bulb and the scale reading in water indicated the specific gravity of the sample.

Figures 4, 5, and 6 illustrate modern conventional forms of the so-called "plain" hydrometer, showing various means for enclosing the ballast of either mercury, metal shot, or sealing wax with shot.

Figures 7 and 8 illustrate the modern conventional form of thermo-hydrometer with the thermometer scale in the bulb, using either mercury or metal shot and sealing wax as ballast. These are best employed where the liquid is transparent, so that the temperature reading may be observed in the liquid and correction made for reducing the hydrometer reading to standard temperature, usually 60 F. (15.56 C.).

Figure 9 illustrates a modern type of thermo-hydrometer with the thermometer scale in the stem for observing the temperature of opaque liquids for temperature correction as above. The hydrometer scale has been developed out of the stem in the illustration to indicate the usual method of inscription.

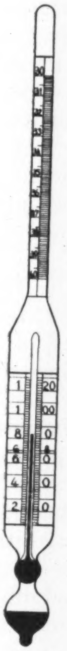

FIG. 7

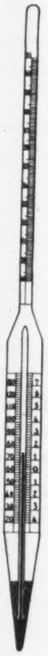

FIG. 8

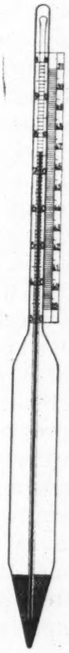

FIG. 9

The basic formulas for relationship of dimensions, volume, and weight in the testing of liquids of various specific gravity are given below:

Assume the volume to the lowest graduation *Su* to equal *V*. The reading at the top graduation is designated as *So* and the total weight of the hydrometer as *W*. Therefore

$$W = V \times \text{specific gravity at } Su \ldots (1)$$

The volume of the stem is expressed by the simple formula for the volume of a cylinder or

$$\frac{\pi d^2 \times l}{4}$$

where:

d = outside diameter of the stem, and

l = length of the stem between *So* and *Su*.

By Archimedes' principle, the weight of the instrument can be equated with the following expression:

$$W = V + \frac{\pi d^2 \times l}{4} \times \text{sp. gr. at } So \ldots (2)$$

therefore from Eqs. 1 and 2

$$V \times \text{sp. gr. at } Su = \left(V + \frac{\pi d^2 \times l}{4}\right) \times \text{sp. gr. at } So$$

$$\text{from which } d = \sqrt{\frac{4V(Su - So)}{\pi l \times So}}$$

$$l = \frac{4 \times V\ (Su - So)}{\pi d^2 \times So}$$

$$V = \frac{\pi d^2\, l \times So}{4(Su - So)}$$

These formulas can be used in constructing an instrument when the desired range and length are given. A skilled craftsmen, however, has no great difficulty in accurately estimating these dimensions from previous experience, when selecting *W* lower body (unterteil) of suitable volume and correct stem diameter for a specified hydrometer.

When standardizing an instrument in manufacture, care must be taken to use a liquid in which the test points are being fixed, which has the same (or as nearly as practical the same) surface tension as the liquid in which the finished hydrometer will be employed. As this is not always feasible in shop practice, as for example in making hydrometers for thick tars or asphalts, a mixture of mobile transparent liquids closely related to the product in this respect are employed.

A typical instance of error is the use of water at the 10° A.P.I. reading. The greater surface tension of water will cause a greater rise above the surface or observation plane around the stem of the hydrometer, thus adding the weight of this water to the hydrometer. This causes the hydrometer to sink lower in water than it would sink in petroleum of the same specific gravity. The finished instrument made this way will result in observations for petroleum which require a plus correction on the A.P.I. scale and a minus correction on the specific gravity scale to balance this error. This difference or correction in indication increases with greater diameter of stem relative to the bulb size when the instrument is tested or calibrated in water (specific gravity at 60 F. = 1.000 or 10° A.P.I.) and observed in petroleum of equal specific gravity.

HYDROMETER SCALES

Specific Gravity or Density Scale:

The normal specific gravity scale assumes water as unity at 60 F. (15.56 C.) readings for the liquid observed or corrected to the same temperature. Usual subdivisions are 0.005 and 0.001. The scale for equal differences in specific gravity is not uniform, as there is an harmonic increase in the distance between subdivisions for equal differences in specific gravity. This can be readily explained by the fact that equal changes in buoyancy cause a greater displacement effect on the instrument, as more of its volume becomes immersed in the liquid. The subdivisions therefore become wider for each scale division in the direction from bottom to top.

It is obvious for this or any other scale that greater sensitivity is obtained by reducing the stem diameter relative to the bulb and *vice versa*. To obtain sensitivity over a large range in a single instrument would result in a very long hydrometer and container, which would be very impractical for several reasons: (*a*) the instrument would be very fragile in handling, (*b*) it would not be stable, if the plane of flotation were near the lower portion of the stem, (*c*) the containers would be correspondingly long and require a considerable volume of liquid for test. It is therefore common practice to make a series of instruments of equal displacement with increase in ballast weight so that the scales of each successive instrument just overlap. It is very seldom that instruments are made with subdivided scale lengths of over 6 in.

The use of arbitrary even scales, based on specific gravity where the subdivisions are uniform, find extremely extensive application. Naturally, since equal increments of specific gravity are not represented by equidistant subdivisions on the scale, it follows that equidistant uniform subdivisions will not represent directly proportional changes in specific gravity values. To relate any arbitrary scale of this type a formula with a determined or accepted modulus is employed. A modulus is defined as a constant factor of a variable function, which serves to connect this function with a particular system or base.

Baumé Scales:

The Baumé scale was first proposed and used in 1768 by the French chemist, Antoine Baumé. The directions for reproducing this scale were to prepare a solution of 15 per cent by weight of very "pure" and very "dry" sodium chloride in 85 per cent by weight of water at 12.5 C. (54.5 F.). The point to which the hydrometer sank in this solution was marked as 15° and the point to which it sank in distilled water was marked 0°. The space between these two marks was subdivided into 15 equal degrees, and divisions of equal length were extended beyond this point.

For liquids lighter than water the scale was established by using a 10 per cent solution of sodium chloride as 0°, and distilled water for the 10° point. The distance between these points was divided into 10 equal parts and these equal divisions extended above the 10° point.

Deviations or errors in this procedure resulted in variations between instruments produced by different manufacturers. It was therefore a more desirable solution to define and fix the relationship between this scale and the specific gravity scale at some definite temperature in mathematical terms in the form of an equation. The adoption of a definite modulus would permit calculation of the scale if the correct specific gravity is known and *vice versa*.

The equations adopted in the United States are as follows:

For liquids heavier than water,

$$\text{Degrees Baumé} = 145 - \frac{145}{\text{sp. gr. } 60/60\text{F.}} \quad (3)$$

or

$$\text{Specific Gravity at } 60/60\text{F.} = \frac{145}{145 - \text{deg. Baumé}}$$

For liquids lighter than water,

$$\text{Degrees Baumé} = \frac{140}{\text{sp. gr. } 60/60\text{F.}} - 130 \quad (4)$$

or

$$\text{Specific Gravity at } 60/60\text{F.} = \frac{140}{130 + \text{deg. Baumé}}$$

A.P.I. (American Petroleum Institute) Scale:

The A.P.I. scale originally used in the United States was generally described as "Baumé for coal oil." Its basis was the modulus for Baumé light hydrometers, namely 140. In some manner, however, a large number of instruments which were put into use in the petroleum industry were based on a modulus of 141.5. There was some confusion due to the fact that some instrument manufacturers adhered to the 140 modulus while one of the principal manufacturers continued to maintain the 141.5 figure as standard. The Bureau of Standards publications from 1901 to 1916 on U. S. Standard Tables for Petroleum Oil accepted the 140 figure as correct.

To terminate this state of affairs the American Petroleum Institute, the U. S. Bureau of Mines, and the U. S. Bureau of Standards agreed in December, 1921, to accept the modulus of 141.5 for the petroleum industry. This scale was to be known as the A.P.I. scale to distinguish it from the original Baumé scale for light liquids.

$$\text{Degrees A.P.I.} = \frac{141.5}{\text{sp. gr. } 60/60\text{F.}} - 131.5 \quad (5)$$

$$\text{Sp. gr. } 60/60\text{F.} = \frac{141.5}{131.5 - \text{deg. A.P.I.}}$$

Baumé for Sugar:

This is the same as the Baumé scale for heavy liquids and the same modulus (145) is employed and is based on specific gravity at 20/20 C.[4]

Brix Scale (Degrees Brix):

The Brix scale is a percentage scale in which each full scale division from 0 to

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[4] *Technologic Paper No. 115*, Nat. Bureau Standards.

100 corresponds to 1 per cent sucrose in water based on specific gravity of water as 1.0000 at 20/20C.[5] Readings in Brix degrees correspond to the following Baumé and specific gravities:

Degrees Brix or Percentage Sucrose by Weight	Specific Gravity at 20/20 C.	Degrees Baumé (modulus 145)
0.0	1.000	0.00
10.0	1.03998	5.57
20.0	1.08287	11.10
30.0	1.12898	16.57
40.0	1.17853	21.97
50.0	1.23174	27.28
60.0	1.28873	32.49
70.0	1.34956	37.56
80.0	1.41421	42.47
90.0	1.48259	47.20
100.0	1.55454	51.73

Twaddle Hydrometer:

The Twaddle scale is related to specific gravity by the following formula:

$$\text{Degrees Twaddle} = \frac{\text{sp. gr. } 60/60\text{F.} - 1.000}{0.005}$$

This scale is sometimes employed in the tanning and textile industries.

Barkometer Scale:

The Barkometer scale is related to specific gravity by the following formula:

$$\text{Specific Gravity} = \text{Barkometer degrees} \times 0.001 + 1.000$$

This scale is likewise used in the tanning industry.

Milk Scales:

1. *Quevenne Lactometer.*—The Quevenne scale is related to specific gravity by exactly the same formula as the Barkometer.

2. *Lactometer Degrees* (*N. Y. Board of Health*).—The N. Y. Board of Health scale is generally accepted as having the following relationship

$$\text{Specific Gravity} = 1 + 0.00029 \times \text{lactometer degrees}$$

Salt Hydrometers:

Salt hydrometers are usually scaled for convenience in percentage or in even arbitrary degrees which are related to specific gravity. To check their accuracy, accepted solution tables should be consulted. These tables give relative solution strength and specific gravity for the particular salt. The following scales are recognized:

1. *Salinometer* (*Sea Water*).—The Salinometer scale is one in which each of 32 subdivisions equals 1 per cent by weight of saline matter in sea water at 200 F.

2. *Salt Hydrometer* (*Saturation Scale*).—This instrument indicates percentage saturation of dry salt dissolved in distilled water, and is, of course, graduated from 0 to 100 per cent.

3. *Salometer for Salt* (*Sodium Chloride*).—The salometer (sodium chloride) is scaled so that each subdivision is equal to $\frac{1}{4}$ per cent dry salt by weight in water at 60 F.

4. *Salometer* (*Calcium Chloride*).—(*a*) The salometer (calcium chloride) scale is divided in degrees with each degree corresponding to $\frac{1}{4}°$ Baumé heavy. Usually scaled from 0 to 120° (0 to 30° Baumé heavy).

(*b*) Also scaled in percentage of dry calcium chloride in water usually 0 to 28 per cent.

NOTE.—Either of the above salometer (calcium chloride) scales is occasionally made with corresponding freezing temperatures for the concentration indicated along various points on the scale.

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[5] There is now no clear distinction between Brix and Balling. Both are interpreted as meaning percentage of sucrose, by weight, at a stated temperature.

Alcohol Scales:

Proof Scale for Ethyl Alcohol.—The proof scale is graduated in *double* the percentage of alcohol by volume (at 60 F.), so that 0 on the scale is distilled water, 100 is 50 per cent by volume of absolute alcohol (in the mixture) to 50 per cent of water, etc. This is also known as the U. S. Internal Revenue Alcohol scale (Per cent Proof Spirits).

A modification of this scale in the U. S. Custom House scale. It has the same basis except that it has 100 subdivisions on each side of a zero corresponding to the same scale as above. The zero is 100 proof or 50 per cent alcohol by volume (at 60 F.). Thus a spirit is rated as *over* proof up to 100 per cent reading from zero up to the top 100 and down to 100 per cent *under* proof reading from zero down to the bottom 100.

Gay Lussac or Tralle:

The Gay Lussac or Tralle scale reads directly in percentage alcohol by volume at 60 F. from zero (water), to 100 per cent (absolute ethyl alcohol). Any indication on this hydrometer would read exactly half of the proof scale in the same strength alcohol solution.

Richter Scale (Per cent of Alcohol by Weight):

The Richter scale would of course agree with the previous one at 0 and 100 per cent. Lower scale indications are obtained with this hydrometer in solutions of equal density and alcohol concentration as compared with the Gay Lussac or Tralle scale for readings between 0 and 100 per cent. Greatest differences (approx. 7.5 per cent) occur in the center of the scale and diminish to zero toward the two extremes of 0 and 100 per cent.

SOIL HYDROMETERS (A.S.T.M. METHOD D 422-39)[6]

A rather unusual application of the hydrometer is in the examination of the density of a suspension of soil particles in water. One hydrometer, (type A) is graduated in a scale of 0 to 60 for observation at 67 F. The 0 corresponds to clear distilled water at 67 F. and each subdivision up to 60 represents grams of suspended soil particles per liter, also at 67 F. The 30 and 60-g. points correspond approximately to a specific gravity of 1.020 and 1.0375, respectively.

The type B hydrometer which is optional for use in this test is a specific gravity hydrometer graduated from 1.000 to 1.050 graduated for reading at 67 F. and is employed in the same manner as the above. Tables and formulas and temperature corrections have been established to correlate the two instruments.

PART II—CALIBRATION OF HYDROMETERS

PREPARED BY E. L. PEFFER

Hydrometers are calibrated by the use of (1) the method of hydrostatic weighing, or (2) by direct comparison with a standard hydrometer.

1. The corrections to the standard hydrometer are determined in the appropriate test liquid the density of which has been determined by the hydrostatic weighing method. In this method a sinker or plummet of known mass and volume is weighed in the test liquid whose density is to be measured, and the density is calculated from the known volume and the difference between the weight of the sinker in vacuum and when immersed in the test liquid.

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[6] Standard Method of Mechanical Analysis of Soils (D 422 - 39), 1939 Book of A.S.T.M. Standards, Part II, p. 453.

Standard hydrometers should be tested at a sufficient number of points so that a smooth calibration curve can be drawn through the points tested.

2. By direct comparison with a standard hydrometer. The procedure is to compare the indication of the instrument under test with that of a standard instrument placed in the same liquid, the correction to be applied to the indication of the standard instrument being known.

Hydrometer scales, as pointed out by J. P. Bader,[7] are graduated in a wide variety of units: for example, specific gravity, density, percentage by weight, and percentage by volume of various substances, and in several independent arbitrary scales.

In general, each hydrometer can be most conveniently tested by direct comparison with a standard graduated in terms of the same unit as that in which the instrument to be tested is graduated; that is, specific gravity instruments by comparison with a standard graduated in terms of similar specific gravity units; percentage alcohol by volume by comparison with a standard graduated in terms of percentage alcohol by volume; degrees A.P.I. by comparison with a standard graduated in terms of degrees A.P.I.; etc.

The standard should be at least as sensitive as the instrument to be tested by comparison with it.

Hydrometers for general laboratory or industrial purposes should be tested at not less than three points.

Cleaning Hydrometers Before Test:

Hydrometers, before test, should be carefully washed with soap and water, and the stems should be wiped with a clean cloth "wet" with alcohol. Extra care must be taken in the cleaning of hydrometers that indicate low percentages of materials having relatively high surface tension. Any contamination of the hydrometer stem or of the liquid surface results in a large error in indication of the instrument.

At the National Bureau of Standards all hydrometers except those for use in oils (which have low surface tension) are tested in a special hydrometer comparator so arranged that the test liquid can be made to overflow immediately before each reading. In this way a fresh, clean liquid surface is in contact with the hydrometer stem at the time each reading is made.

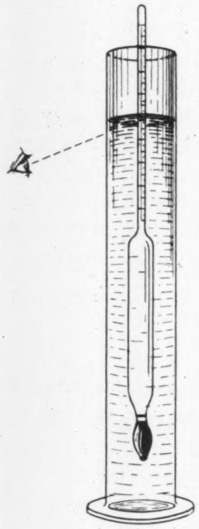

FIG. 10

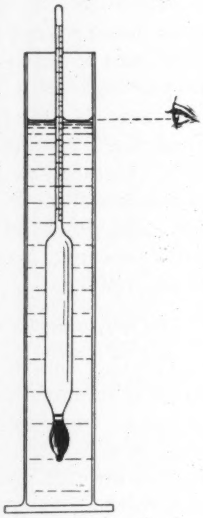

FIG. 11

Method of Reading Hydrometers:

The correct method of reading a hydrometer is illustrated in Figs. 10 and 11. In taking the reading the eye should be placed slightly below the plane of the surface of the liquid (Fig. 10) and then raised slowly until this surface, seen as an ellipse, becomes a straight line (Fig. 11). The point at which this line cuts the hydrometer scale should be taken as the reading of the instrument.

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[7] See p. 466.

In case the liquid is not sufficiently clear to allow the reading to be taken as described above, it will be necessary to read from above the surface and to estimate as accurately as possible the point to which the liquid rises on the stem. It should be remembered, however, that the instrument is calibrated to give correct indications when read at the principal surface of the liquid. It will be necessary, therefore, to correct the reading at the upper meniscus by an amount equal to the height to which the liquid creeps up on the stem of the hydrometer. The amount of this correction may be determined with sufficient accuracy for most purposes by taking a few readings on the upper and the lower meniscus in a clear liquid and noting the differences. The magnitude of the correction will depend upon the surface tension of the liquid, and the length and value of the subdivisions of the hydrometer scale and must be determined in each case for the particular hydrometer and liquid in question.

Hydrometer readings taken at the top of the meniscus in opaque liquids are corrected to the principal surface of the liquids as follows:

The correction for height of meniscus is added to hydrometer readings whenever an increase in the density of the liquid produces a numerical increase in the scale reading (specific gravity, percentage by weight of sugar, and Baumé heavy hydrometers).

The correction is subtracted from hydrometer readings whenever an increase in the density of the liquid produces a numerical decrease in the scale reading (degrees A.P.I. and Baumé light).

Precautions:

Certain precautions must be taken in order that satisfactory results may be obtained. The hydrometer should not only be clean but also dry and at the temperature of the liquid before immersion. The temperature and density of the test liquid must be uniform throughout the cylinder; this is obtained by the use of a stirrer that reaches to the bottom of the container. Readings should not be taken until the hydrometer, which is slowly immersed slightly beyond the test point, floats freely. Both the hydrometer and the liquid should be free from bubbles before the reading is taken.

Test Liquid and Surface Tension:

In the testing of hydrometers it is essential that the test liquid have the same characteristics as that in which the instruments are to be used. This precaution is necessary also in the manufacture (pointing) of hydrometers.

Distilled water at the temperature of standardization may be used for the 1.000 specific gravity point and for the 0 point on alcoholometers and the 0 per cent by weight of sugar. On mineral oil hydrometers, distilled water must not be used as the test liquid for the 1.000 specific gravity point nor the 10° A.P.I. point, at the standard temperature. An oil must be used, so that the surface tension effect will be on the same basis throughout the entire gravity range for petroleum products.

Lactometers, hydrometers for milk, are generally tested in a sulfuric acid solution and the necessary correction made for the difference in the surface tension of milk and the acid.

The magnitude of the correction due to surface tension depends upon the diameter of the stem and the mass of the hydrometer. Hydrometers of equivalent dimensions may be compared, without error, in a liquid differing in surface tension from the specified liquid. Comparisons of dissimilar instruments in such a liquid must be corrected for the effect of the surface tension.

Thermo-Hydrometers:

In certain cases, hydrometers are fitted with thermometers, designed as thermo-hydrometers. The thermometer scale may be either in the body or in the stem of the hydrometer. If within the stem, it should be above the hydrometer scale. In the use of thermo-hydrometers, the instrumental corrections should be determined for the thermometer scale as well as for the hydrometer scale. In reading a thermometer accurately, care must be taken that the line of sight is perpendicular to the thermometer scale in order to avoid parallax.

REFERENCES

(1) "United States Standard Baumé Hydrometer Scales," *Circular No. C59*, Nat. Bureau Standards, April 5, 1916, 13 pp. (Publication is out of print.)

(2) Frederick J. Bates and H. W. Bearce, "New Baumé Scale for Sugar Solutions," *Technologic Paper No. 115*, Nat. Bureau Standards, October 30, 1918, 11 pp.

(3) "United States Standard Tables for Petroleum Oils," *Circular No. 57*, Nat. Bureau Standards, First Edition, January 29, 1916; Second Edition, May 11, 1916; 64 pp. (Superseded by *Circular No. 154*.)

(4) "National Standard Petroleum Oil Tables," *Circular No. 154*, Nat. Bureau Standards, 175 pp. (Superseded by *Circular C410*.)

"Abridged Volume Correction Table for Petroleum Oils," *Supplement to Circular No. 154*, Nat. Bureau Standards, October 8, 1925, 4 pp.

"Abridged Volume Correction Table for Petroleum Oils," *Supplement to Circular No. 154*, Nat. Bureau Standards, October 27, 1931, 4 pp.

"Abridged Volume Correction Table for Petroleum Oils," *Supplement to Circular No. 154*, Nat. Bureau Standards, September 17, 1934, 8 pp. (Superseded by *Supplement to Circular C410*.)

(5) "National Standard Petroleum Oil Tables," *Circular C410*, Nat. Bureau Standards, March 4, 1936, 175 pp.

"Abridged Volume Correction Table for Petroleum Oils," *Supplement to Circular C410*, Nat. Bureau Standards, April 20, 1937, 8 pp.

(6) J. Domke and E. Reimerdes, "Handbuch der Aräometrie," Julius Springer, Berlin (1912).

REPORT OF COMMITTEE E-2
ON
SPECTROGRAPHIC ANALYSIS

Committee E-2 on Spectrographic Analysis held one meeting during the year in Chicago, Ill., on June 26, 1941.

Four new members and two additional member representatives were added to the committee, making the total membership 66. The present officers of the committee were re-elected for the ensuing term of two years.

The committee has in preparation an extension to the "Index to the Literature on Spectrochemical Analysis" to cover the years 1940 and 1941.

Subcommittee III on Quantitative Methods and Applications (M. L. Fuller, chairman) met at Chicago, Ill., on June 26, 1941. The subcommittee has been active during the year through its Groups 1, 2, and 4.

Group 1 on copper, nickel and their alloys (P. A. Leichtle, chairman) is studying methods for the analysis of brass and is making cooperative tests on selected standard alloys.

Group 2 on lead, tin, antimony, bismuth and their alloys (A. E. Ruehle, chairman) has developed proposed Methods for the Spectrochemical Determination of Impurities in Lead Alloys and for the Spectrochemical Determination of Impurities in Tin Alloys. The group has also prepared proposed Specifications for Spectrochemical Apparatus and Techniques. These were submitted to letter ballot and forwarded to Subcommittee III for action. Cooperative tests were made by two of the members on a standard sample of solder, using the method of analysis prepared by the group. Excellent agreement was observed between spectrographic and chemical analysis.

Group 4 on zinc and cadmium and their alloys (M. L. Fuller, chairman) made cooperative tests on a zinc alloy die-casting sample, employing the Tentative Method of Quantitative Spectrochemical Analysis of Zinc Alloy Die Castings for Minor Constituents and Impurities (E 27 – 40 T). The results showed good agreement in general, but certain disagreements will require further work for explanation. Revisions of Tentative Method E 27 – 40 T and of the Tentative Method of Quantiative Spectrochemical Analysis of Zinc for Lead Iron, and Cadmium (E 26 – 35 T), which are under the jurisdiction of Group 4, have been prepared, approved by letter ballot, and forwarded to Subcommittee III for action. Studies are being made of a method for the analysis of cadmium and a spark method for the analysis of zinc.

Subcommittee V on Standards and Pure Materials (T. A. Wright, chairman) has been active throughout the year in answering numerous requests for information on sources of standards. A program is being initiated for the establishment of standard samples of metals and alloys. The subcommittee will prepare recommendations as to sizes and shapes, preparation and analysis, and will act as a clearing house for information on these materials.

This report has been submitted to letter ballot of the committee which consists of 66 members; 50 members returned their ballots, of whom 46 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. V. CHURCHILL,
Chairman.

B. F. SCRIBNER,
Secretary.

REPORT OF COMMITTEE E-3

ON

CHEMICAL ANALYSIS OF METALS

Committee E-3 on Chemical Analysis of Metals held a meeting in Chicago, Ill., on June 25, 1941, during the annual meeting of the Society. Five division and thirteen subcommittee meetings were also held during the year.

Six new members were elected during the year. The committee wishes to record its sorrow in the loss by death of J. B. Mosley, chairman of Subcommittee B-2. His interest in the work of Committee E-3, and his friendly personality will be missed.

Notwithstanding the demands of the war period, considerable progress has been made by the different division and subcommittee groups.

Methods for the analyses of pig lead, nickel-chromium electrical-heating alloys, slab zinc, and brasses have been approved by letter ballot of the committee and are being submitted to the Society as new tentative standards.

Subsequent to the annual meeting, Committee E-3 presented to the Society, through Committee E-10 on Standards, a tentative revision of the Standard Methods of Chemical Analysis of Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron (E 30 – 39). This tentative revision, in the form of new Tentative Methods of Chemical Analysis for Aluminum, Columbium, and Lead in Steel (E 30 – 41 T), was accepted[1] by Committee E-10 on October 22, 1941, and has been published separately as a 1941 Supplement to the Methods of Chemical Analysis of Metals.

I. NEW TENTATIVE STANDARDS

The committee recommends for publication as tentative, the following new methods of chemical analysis as appended hereto[2] to replace the several present methods as indicated:

Methods of:

Chemical Analysis of Magnesium and Magnesium Alloys,
Chemical Analysis of Aluminum and Aluminum Alloys, to replace Tentative Methods B 40 – 36 T,
Chemical Analysis of Brasses, to replace Tentative Methods B 45 – 36 T,
Chemical Analysis of Pig Lead, to replace Tentative Methods B 35 – 36 T,
Chemical Analysis of Metallic Materials for Electrical Heating, to replace Tentative Methods B 71 – 34 T,
Chemical Analysis of Nickel, to replace Tentative Methods B 41 – 36 T, and
Chemical Analysis of Slab Zinc (Spelter), to replace Tentative Methods B 38 – 36 T.

II. ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the following three methods be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Methods of:

Chemical Analysis for Aluminum, Columbium, and Lead in Steel (E 30 – 41 T),[3] to be incorporated in Standard E 30 – 39,[4]

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[1] In submitting this recommendation to Committee E-10 on Standards, Committee E-3 reported results of the letter ballot vote as follows: Of a total membership of 70, 59 members returned their ballots, of whom 49 voted affirmatively, 1 negatively, and 9 members marked their ballots "not voting" with respect to determination of aluminum; 46 voted affirmatively, 0 negatively, and 13 members marked their ballots "not voting" with respect to determination of columbium; 50 voted affirmatively, 0 negatively, and 9 members marked their ballots "not voting" with respect to determination of lead.

[2] These methods were accepted as tentative by the Society and appear in the A.S.T.M. Methods of Chemical Analysis of Metals, 1942.

[3] 1941 Supplement to Chemical Analysis of Metals.

[4] A.S.T.M. Methods of Chemical Analysis of Metals, 1939, pp. 1, 63, 54.

Chemical Analysis of Ferro-Alloys (E 31 - 38 T),[3] and
Sampling Ferro-Alloys (E 32 - 39 T).[4]

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 68 members, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Item	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Chemical Analysis of Magnesium and Magnesium Alloys..	50	0	7
Chemical Analysis of Aluminum and Aluminum Alloys, to replace Tentative Methods B 40 - 36 T..................	47	0	7
Chemical Analysis of Brasses, to replace Tentative Methods B 45 - 36 T..............	55	1	7
Chemical Analysis of Pig Lead, to replace Tentative Methods B 35 - 36 T..................	50	0	10
Chemical Analysis of Metallic Materials for Electrical Heating, to replace Tentative Methods B 71 - 34 T........	49	0	11
Chemical Analysis of Nickel, to replace Tentative Methods B 41 - 36 T..................	52	0	3
Chemical Analysis of Slab Zinc (Spelter), to replace Tentative Methods B 38 - 36 T....	49	0	11
II. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Chemical Analysis for Aluminum, Columbium, and Lead in Steel (E 30 - 41 T), to be incorporated in Standard E 30 - 39....................	50	0	8
Chemical Analysis of Ferro-Alloys (E 31 - 38 T)........	51	0	7
Sampling Ferro-Alloys (E 32 - 39 T)......................	52	0	6

ACTIVITIES OF DIVISIONS AND SUBCOMMITTEES

Division A on Ferrous Metals (Arba Thomas, chairman).—Work is in progress on methods for the determination of small amounts of lead in steel, aluminum in alloy steels, and selenium in stainless steels.[4] Subcommittees have been formed to study methods for the determination of nitrogen (W. D. Brown, chairman), bismuth (W. J. Boyer, chairman), and tellurium (R. H. Elder, chairman).

Division B on Non-Ferrous Metals (D. R. Evans, chairman).—The division is actively continuing the revision of the present A.S.T.M. methods for the analysis of non-ferrous alloys.

Subcommittee B-1 on Copper and Its Alloys (R. P. Nevers, chairman) has completed work on the analytical procedures for brasses and is now engaged in developing methods for the analysis of bronzes.

Subcommittee B-2 on Lead, Tin, Antimony, Bismuth, and Their Alloys (C. A. Ray, chairman) has prepared methods for the analysis of solder which have been tested on a specially prepared sample of solder (35 per cent tin, 65 per cent lead). It is expected that the methods will be submitted to the division during the latter part of the year.[4]

Subcommittee B-3 on Aluminum and Magnesium and Their Alloys (H. V. Churchill, chairman) developed the methods for the analysis of aluminum and magnesium and their alloys being submitted for publication as tentative as mentioned earlier in this report.

Subcommittee B-4 on Zinc and Its Alloys (R. W. Coltman, chairman) has prepared methods for the analysis of zinc-base alloys. The methods are now being tested prior to submittal to Division B.

Subcommittee B-6 on Nickel (Calvin Sterling, chairman) is studying methods for the analysis of nickel which it prepared.

Division D on General Analytical Methods (B. L. Clarke, chairman).—The division has begun the preparation of a section on apparatus and standard solutions for the next issue of the publication of A.S.T.M. Methods of Chemical Analysis of Metals. The work is being done by Subcommittee D-1 on Development, Preparation, or Standardization of Reagents (S. E. Q. Ashley, chairman), and Subcommittee D-2 on Development and Standardization of Apparatus (E. B. Rubloff, chairman).

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[4] See Editorial Note, p. 477.

The present officers of the committee were re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 68 members; 62 members returned their ballots, of whom 60 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

G. E. F. LUNDELL,
Chairman.

H. A. BRIGHT,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee E-3 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative Methods for:

Chemical Analysis of Tin- and Lead-Base Solder Metal (E 46 – 42 T), to replace immediately the Tentative Methods of Chemical Analysis of Alloys of Lead, Tin, Antimony, and Copper (B 18 – 36 T), and
Chemical Analysis of Zinc-Base Die Casting Alloys (E 47 – 42 T).

These recommendations were accepted by Committee E-10 on December 11, 1942, and assigned the A.S.T.M. serial designations indicated above. The new tentative standards appear in the "A.S.T.M. Methods of Chemical Analysis of Metals (1942)."

A new Method of Chemical Analysis for Determining Selenium in Steel (E 30 – 43 T) was accepted by Committee E-10 on March 6, 1943, as a tentative revision of the Standard Methods of Chemical Analysis of Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron (E 30 – 42). This also appears in the "A.S.T.M. Methods of Chemical Analysis of Metals (1942)."

On March 6, 1943, Committee E-10 accepted the recommendation of Committee E-3 that the following A.S.T.M. tentative methods, which are now out-of-date, be withdrawn:

Tentative Methods of:

Chemical Analysis of Manganese Bronze (B 27 – 36 T),
Chemical Analysis of Gun Metal (B 28 – 36 T), and
Chemical Analysis of Bronze Bearing Metal (B 46 – 36 T).

REPORT OF COMMITTEE E-4

ON

METALLOGRAPHY

During the past year, Committee E-4 on Metallography held an informal meeting in Cleveland, Ohio, on March 4, 1942, and its regular meeting in Atlantic City, N. J., on June 24, 1942, during the annual meeting of the Society.

Recommendations on several new and revised tentative standards were considered at the June meeting of the committee and action taken to submit these to letter ballot as referred to later in this report.

At the June meeting, the present officers of the committee were re-elected for the ensuing term of two years.

Committee E-4 reports the loss of one member during the year leaving a total of 67 members.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Selection and Preparation of Samples (G. F. Comstock, chairman).—During the year this subcommittee has made a splendid contribution by making an extensive revision of the Tentative Methods of Preparation of Metallographic Specimens (E 3 – 39 T) which extends the subject matter to include more advanced practices covering electrolytic polishing of metallographic specimens. It is expected that this recommendation will be ready for submittal to the Society through Committee E-10 on Standards later in the year.[1]

Subcommittee IV on Photography (L. V. Foster, chairman).—This subcommittee has carefully reviewed the Tentative Methods of Preparation of Micrographs of Metals and Alloys (E 2 – 39 T) and recommends that they be continued as tentative without revision.

Subcommittee VI on X-ray Methods (W. L. Fink, chairman).—The Joint Committee on Chemical Analysis by X-ray Diffraction Methods has prepared a 4000-card index of X-ray diffraction data for use in the Hanawalt method of chemical analysis by X-ray diffraction. Copies of the Card Index File are available from A.S.T.M. Headquarters at $50.00 per set.

A proposed Recommended Practice for Identification of Crystalline Materials by the Hanawalt X-ray Diffraction Method which covers in detail the test procedure to be followed has been completed by this subcommittee and will be presented to the Society through Committee E-10 on Standards subsequent to the annual meeting.[1]

Subcommittee VII on Dilatometry (L. H. Carr, chairman).—A recommended practice for dilatometric analysis is now in the process of final revision and editing prior to submittal to letter ballot. It is expected to be ready before the next meeting of Committee E-4.

Subcommittee VIII on Grain Size (M. A. Grossman, chairman).—Inasmuch as no criticisms of the Tentative Classification of Austenite Grain Size in

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[1] See Editorial Note, p. 479.

Steels (E 19 - 39 T) have been received, the subcommittee recommends that this classification be continued as tentative without revision.

The subgroups concerned with ferrite grain size and with non-ferrous grain size are both actively engaged in preparing certain recommended changes which will result in improvements in the grain size reference standards.

Subcommittee IX on Inclusions (Samuel Epstein, chairman).—This committee has had an extremely active year, having held a number of meetings, and has completed a proposed Tentative Recommended Practice for Determining the Inclusion Content of Steel. Due to urgent requests that this recommended practice be made available this year, the subcommittee has made every effort to complete the final details of the procedures to be included. It is expected that these recommendations will be ready for presentation to the Society through Committee E-10 subsequent to the annual meeting.[1]

Respectfully submitted on behalf of the committee,

L. L. WYMAN,
Chairman.

J. J. BOWMAN,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee E-4 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative Recommended Practice for:
Identification of Crystalline Materials by the Hanawalt X-Ray Diffraction Method (E 43 - 42 T),
Determining the Inclusion Content of Steel (E 45 - 42 T), and

Revision of Tentative Methods of:
Preparation of Metallographic Specimens (E 3 - 39 T).

These recommendations were accepted by Committee E-10 on November 27, 1942, and the new and revised tentative standards appear in the 1942 Book of A.S.T.M. Standards, Part I, pp. 1537, 1543, and 1586.

REPORT OF COMMITTEE E-8

ON

NOMENCLATURE AND DEFINITIONS

While Committee E-8 on Nomenclature and Definitions has not held a meeting during the past year, a number of matters have been handled by correspondence.

Under the rotational basis on which the Advisory Committee is now functioning with the terms of two members expiring each year, the Executive Committee of the Society has reappointed P. V. Faragher and L. H. Fry for a three-year term, expiring in 1945.

A continuing interest in the establishment of authoritative definitions and nomenclature relating to engineering materials is evident from the following new and revised definitions being presented by the standing committees to the Society, for publication as tentative, at this annual meeting:

Tentative Definitions of Terms Relating to Concrete and Concrete Aggregates (C 125 – 36 T), new definitions of "flat piece," "elongated piece," "water-cement ratio," and revised definition of "blast furnace slag," prepared by Committee C-9 on Concrete and Concrete Aggregates,

Tentative Definitions of Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16 – 41 T), new definitions of "lacquer," "sanitary or tin plate lacquers," "putty," "bituminous varnish," "glaze," and "surfacer," prepared by Committee D-1 on Paint, Varnish, Lacquer, and Related Products,

Tentative Method of Test for Spectral Characteristics and Color of Objects and Materials, prepared by Committee D-1, includes descriptions of terms and symbols,

Tentative Methods of Test for Neutralization Number of Petroleum Products, prepared by Committee D-2 on Petroleum Products and Lubricants, includes descriptions of terms,

Definitions of Terms Relating to Soaps and Other Detergents (D 459 – 41 T), prepared by Committee D-12 on Soaps and Other Detergents,

Tentative Classification of Terms and Descriptive Nomenclature of Objects Made from Plastics, prepared by Committee D-20 on Plastics, and

Tentative Recommended Practice for Repeated Flexural Stress (Fatigue) Test of Plastics, prepared by Committee D-20.

Committee D-4 on Road and Paving Materials has referred to Committee E-8 some fifteen definitions of terms relating to bituminous materials which represent a revision of and includes several new definitions for inclusion in the Standard Definitions of Terms Relating to Materials for Roads and Pavements (D 8 – 33). Also Committee D-8 on Bituminous Waterproofing and Roofing Materials has referred to Committee E-8 definitions of seven terms relating to waterproofing and roofing materials. These proposed definitions have been referred for review to the Subcommittee on Definitions Relating to Bituminous Materials under the chairmanship of K. G. Mackenzie.

Committee E-8 at its last meeting authorized the organization of a special Subcommittee on Definition of the Term Glass for the purpose of reconciling the differences between the Tentative Definition of the Term Glass (C 162 – 41 T), prepared by Committee C-14 on Glass and Glass Products, and the definition for glass prepared by Committee D-13 on Textile Materials and included in the Definitions of Terms Relating to Textile Materials (D 123 – 41). Invitations were extended to the interested standing committees of the Society to designate representatives to serve on this subcommittee and the following have been appointed:

W. F. Bartoe, Representing Committee D-20 on Plastics,

F. C. Flint, Representing Committee C-14 on Glass and Glass Products,

J. C. Hostetter, Representing Committee C-14,
J. W. McBurney, Representing Committee C-15 on Manufactured Masonry Units,
J. L. Miner, Representing Committee C-1 on Cement,
E. M. Penney, Representing Committee D-13 on Textile Materials,
John Sabo, Representing Committee D-9 on Electrical Insulating Materials, and
G. N. Thompson, Representing Committee C-5 on Fire Tests of Materials and Construction.

A meeting of the special Subcommittee on Glass was held in Philadelphia, Pa., on May 15, 1942, at which time the two existing definitions were carefully reviewed and many valuable suggestions offered. Action was taken to refer this matter to Subcommittee I on Nomenclature and Definitions of Committee C-14 with the thought that that committee would formulate the technical part of the definition in the light of the discussion at this meeting, in consultation with Committee D-13. Any note calling attention to organic glasses would be referred to Committee D-20 for review.

Committee D-9 on Electrical Insulating Materials has referred to Committee E-8 for review the following definition of a generic name for a group of nonoil liquid insulating compounds:

Askarel.—A synthetic noninflammable insulating liquid which, when decomposed by the electric arc, evolves only nonexplosive gaseous mixtures.

It is understood that this definition has been adopted by the National Electrical Manufacturers Assn. and is under consideration in Committee D-9 in connection with its work on insulating oils.

The Subcommittee on Definition of Terms Relating to Heat Treatment (L. H. Fry, chairman) has reviewed the Definitions of Terms Relating to Heat Treating[1] prepared by the Joint Committee on Definitions of Terms Relating to Heat Treatment, comprising representatives of the four sponsoring societies, namely, the American Society for Metals, the Society of Automotive Engineers, the American Foundrymen's Assn., and the American Society for Testing Materials. The subcommittee has approved the definitions for publication and it is planned to submit these definitions to letter ballot in Committee E-8 for presentation to the Society for publication as tentative, subsequent to the annual meeting through Committee E-10 on Standards.

I. ADOPTION OF TENTATIVE STANDARD AS STANDARD

On the recommendation of the Subcommittee on Sieve and Screen (Cloyd M. Chapman, chairman), the committee recommends that the Tentative Definition of the Term Screen (Sieve) (E 13 - 28 T) be approved for reference to letter ballot of the Society for adoption as standard with a revision in Note 2 changing "rectangular" to read "square." With this change, the definition reads as follows:

Screen (Sieve).—A plate or sheet or a woven cloth, or other device, with regularly spaced apertures of uniform size, mounted in a suitable frame or holder, for use in separating material according to size.

NOTE 1.—The shape and spacing of apertures, size of wires or threads, thickness of plate or sheet, allowable variations and similar properties should be taken care of in specifications.

NOTE 2.—In mechanical analysis testing work, when not otherwise specified, the term "sieve" shall apply to an apparatus in which the apertures are square, and the term "screen" shall apply to an apparatus in which the apertures are circular.

The results of the vote in Committee E-8 on this recommendation are as follows: From a membership of 52, 37 members returned their ballots, of whom 27 voted affirmatively, 0 negatively, and 10 members marked their ballots "not voting."

II. REVIEW OF DEFINITIONS RECOMMENDED FOR ADOPTION AS STANDARD

The following actions were taken by the committee on tentative definitions being recommended for adoption as standard by other Society committees at this annual meeting:

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[1] See Editorial Note, p. 482.

Refractories.—The committee has reviewed during the year the Tentative Definitions of Terms Relating to Refractories (C 71 – 40 T). These definitions had been recommended by Committee C-8 on Refractories for adoption last year and Committee E-8 requested that this action be deferred for a year in order that certain editorial suggestions might be reviewed and discussed with Committee C-8. This review developed several suggestions which were presented to and found acceptable by Committee C-8. These editorial changes comprised principally the addition of the words "of refractories" to the names of the terms defined and in the definition of erosion, it was suggested that the phrase "such as molten metals and slags" be omitted. With these editorial changes, the definitions of the terms abrasion, burning, corrosion, erosion, and slagging have been approved by Committee E-8 for submittal to the Society for adoption as standard by letter ballot vote as follows: From a membership of 52, 35 members returned their ballots, of whom 31 voted affirmatively, 0 negatively, and 4 members marked their ballots "not voting."

Gypsum.—Committee C-11 on Gypsum is recommending for adoption as standard this year the following revised definition of the term "perforated gypsum lath":

Perforated Gypsum Lath.—A gypsum lath with perforations. For one hour fire resistive construction, perforated gypsum lath shall be not less than $\frac{3}{8}$ in. in thickness with perforations not less than $\frac{3}{4}$ in. in diameter with one perforation for not more than each 16 sq. in. of lath.

This proposed revised definition was reviewed by Committee E-8 and action taken to request Committee C-11 to defer adoption[2] of it for a year in order to provide opportunity for discussion of desirable changes.

Rheology.—The Tentative Definitions of Terms Relating to Rheological Properties of Matter (E 24 – 40 T) are being recommended for adoption as standard this year by Committee E-1 on Methods of Testing. These definitions have been reviewed by Committee E-8 and the comments received will be referred to Committee E-1 for consideration.

This report has been submitted to letter ballot of the committee which consists of 52 members; 25 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

CLOYD M. CHAPMAN,
Chairman.

R. E. HESS,
Ex-officio Secretary.

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[2] See Summary of Proceedings, p. 23.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee E-8 presented to the Society through Committee E-10 on Standards proposed Tentative Definitions of Terms Relating to Heat Treatment of Metals. These definitions were accepted by Committee E-10 for publication as tentative on August 24, 1942, to replace immediately the Standard Definitions of Terms Relating to Heat Treatment Operations (Especially as Related to Ferrous Alloys) (A 119 – 33). The new tentative definitions were assigned the designation E 44 – 42 T and appear in the 1942 Book of A.S.T.M. Standards, Part I, p. 1592.

REPORT OF COMMITTEE E-9
ON
RESEARCH

With this report Committee E-9 on Research reviews briefly certain research activities and matters related thereto which have engaged its attention during the past year. As customary, a more comprehensive review of research activities throughout the Society will appear in a subsequent issue of the ASTM Bulletin.

Research Committee Activities:

The Research Committee on Fatigue of Metals has been conducting a study of size effect on fatigue specimens free from highly localized stress. Material has been secured for tests of an SAE 1020 carbon steel as rolled, an SAE X4130 chromium-molybdenum steel, heat treated, and an SAE 1035 steel, hot rolled. A progress report[1] on this investigation is being presented to the Society at this annual meeting. Committee E-9 has appropriated the sum of $800 from the Society's Research Fund in support of this program.

The Research Committee has asked Committee E-9 for an additional contribution of $1000 from the Research Fund to continue its study of size effect. Committee E-9 believes that this is an important piece of research and has accordingly placed the sum requested at the disposal of the Research Committee.

The Joint Research Committee on Effect of Temperature on the Properties of Metals of The American Society of Mechanical Engineers and this Society is presenting a report[2] at the annual meeting briefly summarizing the progress made during the year in its various research projects. These include tests of tubular members at elevated temperatures, effect of variables on high-temperature properties of metals, comparison of short-time test methods, and a review of the present high-temperature test methods.

The Joint Research Committee on Boiler Feedwater Studies is presenting a progress report to the Society at this annual meeting discussing primarily the research project on caustic embrittlement and referring briefly to other problems that have been considered.

Joint Research Project on Durability of Concrete.—Work on this project is still in a formative stage but it is expected that out of its studies valuable information will develop. Committee E-9 has made a nominal contribution of $250 from the Research Fund to this project to take care of certain administrative expenses.

Research Progress of A.S.T.M. Committees:

A detailed review of research projects being carried out by various A.S.T.M. committees appeared in the January issue of the ASTM Bulletin, which gives reference to the Society publications in which they are described. This review covers primarily two classifications: (*a*) new work initiated or contemplated; and (*b*) activities in which substantial progress has been noted during the year.

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[1] See p. 145.

[2] See p. 154.

The specific research projects given below have been followed in a general way by Committee E-9:

Effect of Pretreatments in Painting of Structural Steel (Committee D-1).—Since the last annual meeting Subcommittee XXIX of Committee D-1 on Paint, Varnish, Lacquer, and Related Products has made two additional inspections of its test panels and it is expected to present the results of these inspections in the annual report of Committee D-1. At the request of Committee E-9, Subcommittee XXIX has listed a number of problems that are being considered for study.

Testing of Textile Finishes (Committee D-13).—The research project on the development of methods for the evaluation of finishes of textile fabrics was concluded in October, 1941, at the end of its second year, and a brief summary of what has been accomplished is covered in the current report[3] of Committee D-13. A Permanent Committee has been appointed to see to the adoption by Committee D-13 of the instruments and methods that have been developed; to answer inquiries that may arise after publication of papers describing the instruments and the methods for their use, and to promote the adoption of the instruments wherever possible; and to renew the research program at any time that it may be indicated. In the latter connection a Program Committee has been appointed to determine the interest of the subscribers in continuing the work into a third year and if the response is favorable to set up a program covering further study of the application and refinement of the instruments developed; also work on the evaluation of crease-resistant, shrink-resistant, and slip-resistant finishes.

Study of Test Methods for Plastics (Committee D-20).—Considerable study has been undertaken in Committee D-20 on Plastics on the problems before it and Committee E-9 has noted that consideration is to be given in the committee to a more systematic approach to these studies.

Study to Determine if Results of Empirical Flow Tests can be Expressed in Absolute Units (Committee E-1).—As mentioned in last year's report, this study is being carried out at Brooklyn Polytechnic Institute.

Organization of A.S.T.M. Research Activities in Corrosion:

During the year, Committee E-9 has arranged several conferences of the chairmen of the A.S.T.M. committees concerned with corrosion problems looking towards the setting up of a more effective means of administering the Society's work in the field of corrosion of metals. These committees are A-5 on Corrosion of Iron and Steel, A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys, B-3 on Corrosion of Non-Ferrous Metals and Alloys, B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys, B-6 on Die-Cast Metals and Alloys, and B-8 on Electrodeposited Metallic Coatings. As a result of these conferences it has been recommended that there be formed a new administrative committee, to be designated as the Advisory Committee on Corrosion. An Outline of Scope of Activities and Functions of the Advisory Committee has been prepared and approved by the Executive Committee of the Society and it is expected that the organization meeting of the Advisory Committee will be held during this annual meeting. Under the provisions of the Outline of Scope of Activities and Functions, the Advisory Committee will consist of the chairmen (or alternate) of the six standing committees named above and three members-at-large, serving for a term of three years, appointed by the Executive Committee of the Society, and will be concerned with corrosion investigations, researches, test procedures, etc., in so far as these apply to metals and alloys. The committee will also serve as an advisory group to the Executive Committee and Committee E-9 with respect to corrosion problems.

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[3] See p. 431.

Proposed Foundation for Scientific Research in Rubber Technology:

Mention was made in the report last year that Technical Committee A on Automotive Rubber, of Committee D-11 on Rubber Products, has under consideration the formation of a Foundation for Scientific Research in Rubber Technology. This matter has been held in abeyance at the suggestion of the special committee developing plans for the proposed research. There is still the feeling among engineers, particularly in the consumer lines, that there is a need for considerably more knowledge than we have on some of the fundamental problems and it is expected that, despite the scarcity of rubber because of the war, provision will be made for test rubber for experimental work. It has been noted that courses of instruction in rubber technology have recently been established in two colleges which will tend to foster research in this field, and this may serve the purpose in the minds of those who originally proposed the research project to Technical Committee A.

Research Activities of Other Societies:

The Society has continued its representation on such groups as the Division of Engineering and Industrial Research and the Highway Research Board of the National Research Council, the A.S.M.E. Research Committee on Mechanical Springs, and the Welding Research Committee of Engineering Foundation. Research activities have been continued along the lines mentioned in previous reports and have been reviewed during the year by Committee E-9.

A.S.T.M. Research Activities Under War Conditions:

As the result of discussions in the Executive Committee of the Society, Committee E-9 has given consideration to the desirable policy for A.S.T.M. research activities under war conditions. Of course, our national research activities must be directed primarily to the development of implements of war, to improving our situation as to raw materials and transportation and to the development of superior processing methods. The Society activity should be directed toward the same ends.

Even though immediate results are demanded for war production, research work will be completed sooner if sufficient time and effort are taken for an analysis of the problem, the utilization of previous research and a careful planning and supervision of the project. Economy of time in the laboratory requires careful analysis and evaluation of data. The full application of research to design and production is contingent upon the proper presentation of the work and distribution of reports.

Within the membership of our research committees and the research groups of standing committees are the foremost authorities of the country. Collectively, our committees have a full knowledge of the published literature and of the vast unpublished data of industrial and independent laboratories. They know the facilities for research, the qualifications of research personnel and the needs for both applied and fundamental research. Our committees operate cooperatively in organizing work and in presenting, checking and reviewing results. They are in intimate contact with industry and possess facilities for dissemination of information.

The quantity of research personnel available is not readily expanded because of the training and aptitude required for successful work. The output of the country's research personnel can be increased through the utilization of agencies equipped to outline without delay logical research programs without unnecessary duplication of work and with a clear understanding of the problem. Our committees are capable of rendering a national service in raising the output of the nation's research organization.

How our committees can be fully utilized deserves consideration. There has been a tendency for the National Government to organize its research activities independent of the cooperative structure evolved by industry and represented by the technical societies. Possibly such action cannot be avoided due to the limitation in the use of Government funds and the requirements of war. But the consequences will be bad if the cooperative research undertakings of our technical societies are weakened or destroyed because of the power of huge federal appropriations.

If our research committees are to survive they must function now during this war in constructive work directed to the national war policy. The National Government needs and, it is believed, will welcome such efforts. Our traditions are democratic and democracy can operate effectively in research as well as in the fields of politics.

There can be no question as to whether research is fundamental. The fundamental approach may consume less time than less well organized hit-and-miss methods. A fundamental knowledge assists both in the interpretation and application of research. It is especially needed in time of war when new untrodden fields must be explored. There can be no limitation fixed because of the time factor. It is beyond human power to predict when this war will end. It is certain, however, that cooperative research efforts directed to the ends of national policy will shorten the period of this world struggle.

It is urged, therefore, as a constructive policy that our research committees and research groups maintain the initiative in organizing, directing, interpreting, and disseminating research activities—all directed toward the war policy.

Administrative Matters:

C. D. Holley, whose term would have expired with this annual meeting, died on January 16, 1942. The appointment of a new member for the term of five years will be made by the Executive Committee of the Society.

The principal of the A.S.T.M. Research Fund is now $32,391.15, an increase of $3000 since the report a year ago. The increase comprises $2600 from 1941 entrance fees (which together with the $600 reported last June from that source represents the entire amount collected in 1941 from entrance fees) and $400 from current (1942) entrance fees.

The balance of income in the Research Fund on May 31, 1942, was $1052.48. This is after payment of $500 of the $1000 appropriated to the Research Committee on Fatigue of Metals and $250 to the Research Project on Durability of Concrete. The present annual available income from the principal of the Fund is about $1125.

Respectfully submitted on behalf of the committee,

G. F. JENKS,
Chairman.

C. L. WARWICK,
Secretary.
June, 1942.

REPORT OF COMMITTEE E-10
ON
STANDARDS

Committee E-10 on Standards has had one of the most active years in its career. This, of course, has been due to the desire on the part of the standing committees, as a part of the war effort, to have published as promptly as possible new standards, revisions of tentative standards, and tentative revisions of standards as well as a number of emergency proposals in order to meet the needs of the present situation. This has meant that the E-10 procedure for approval of tentative standards and revision of existing tentative standards has been used to an extent seldom, if ever, exceeded in past years and added to this has been the consideration and acceptance of over sixty proposals of an emergency nature. These latter fall under the procedure established by the Executive Committee for setting up emergency alternate provisions or complete emergency standards in the interest of expediting procurement or of conserving materials during the period of National Emergency. Under this procedure, emergency alternates, which have been approved by the appropriate subcommittee or subgroup of the sponsoring committee and have the endorsement of the chairman of the main committee, may be submitted to Committee E-10 for approval for publication. Thus far, fifty such emergency provisions, one complete emergency alternate standard, and six emergency standards have been approved by Committee E-10.

In view of this inordinate number of recommendations coming to Committee E-10 for action, two meetings have been held during the year rather than the usual single meeting. These have been held in Philadelphia, Pa. on August 25, 1941, and on April 6, 1942. In addition, a great number of recommendations have been acted upon by letter.

At the August meeting, J. R. Townsend was elected chairman of Committee E-10 to serve until June, 1942. The membership on Committee E-10 of R. P. Anderson and H. S. Mattimore expired last year. The Executive Committee appointed R. D. Bonney of Congoleum-Nairn, Inc. and F. H. Jackson of the Public Roads Administration to serve as members of Committee E-10 for a three-year term, expiring in 1944.

REVIEW OF A.S.T.M. STANDARDIZATION ACTIVITIES

A summary of the new and revised tentative standards submitted at the 1941 annual meeting was included in the August issue of the ASTM BULLETIN together with a review of the standardization work of the various committees, with statements concerning new standards in prospect. Much of the information, particularly with respect to standardization projects under development in committees, is furnished by the standing committees subsequent to the annual meeting and takes advantage of the fact that many committees plan their next year's work at committee meetings held during the annual meeting.

At the present writing, it is possible to make only a preliminary estimate in terms of new and revised standards of the volume of standardization work accomplished during the year and reported to the Society at this meeting. The reports of standing committees to be presented at the annual meeting include recommendations to the Society substantially as follows:

New standard	1
New tentative standards	55
Revisions of existing tentative standards	50
Tentative revisions of existing standards	14
Tentative standards recommended for adoption as standard	63[a]
Revisions of existing standards recommended for adoption as standard	86
Standards and tentative standards withdrawn or replaced	14

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[a] Of these 63 tentative standards being adopted 6 are being added to existing standards.

There are at present 650 standards and 399 tentative standards of the Society, making a total of 1049, and if the above recommendations are approved this total will become 1085.

CONSIDERATION OF NEW AND REVISED STANDARDS

Proposed new standards accepted by Committee E-10 and published as tentative during the year are as follows:

Tentative Specifications for:

Atomic-Hydrogen-Arc-Welded and Electric-Resistance-Welded Alloy-Steel Boiler and Superheater Tubes (A 249 – 41 T), submitted by Committee A-1 on Steel,

Electric-Resistance-Welded Carbon - Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 250 – 41 T), submitted by Committee A-1,

Iron and Steel Gas Welding Rods (A 251 – 42 T), submitted by Committee A-1,

Welded and Seamless Steel Pipe Piles (A 252 – 42 T), submitted by Committee A-1,

Nickel Rods and Bars (B 160 – 41 T), submitted by Committee B-2 on Non-Ferrous Metals and Alloys,

Nickel Cold-Drawn Pipe and Tubing (B 161 – 41 T), submitted by Committee B-2,

Nickel Plate, Sheet, and Strip (B 162 – 41 T), submitted by Committee B-2,

Nickel, Nickel-Copper Alloy, and Nickel-Chromium-Iron Alloy Seamless Condenser Tubes and Ferrule Stock (B 163 – 41 T), submitted by Committee B-2,

Nickel-Copper Alloy Rods and Bars (B 164 – 41 T), submitted by Committee B-2,

Nickel-Copper Alloy Cold-Drawn Pipe and Tubing (B 165 – 41 T), submitted by Committee B-2,

Nickel-Chromium-Iron Alloy Rods and Bars (B 166 – 41 T), submitted by Committee B-2,

Nickel-Chromium-Iron Alloy Cold-Drawn Pipe and Tubing (B 167 – 41 T), submitted by Committee B-2,

Nickel-Chromium-Iron Alloy Plate, Sheet, and Strip (B 168 – 41 T), submitted by Committee B-2,

Oxygen-Free Electrolytic Copper Wire Bars, Billets, and Cakes (B 170 – 42 T), submitted by Committee B-2,

Phosphor Bronze Rods, Bars, and Shapes (B 139 – 41 T), submitted by Committee B-5 on Copper and Copper Alloys, Cast and Wrought,

Phosphor Bronze Wire (B 159 – 41 T), submitted by Committee B-5,

Aluminum Bronze Sheet and Strip (B 169 – 41 T), submitted by Committee B-5,

Copper-Alloy Condenser Tube Plates (B 171 – 42 T), submitted by Committee B-5,

Slow-Setting Emulsified Asphalt (for Fine Aggregate Mixes) (D 631 – 41 T), submitted by Committee D-4 on Road and Paving Materials,

Sodium Chloride (D 632 – 41 T), submitted by Committee D-4,

Molds for Test Specimens of Molding Materials Used for Electrical Insulation (D 647 – 41 T), submitted by Committee D-9 on Electrical Insulating Materials,

Tentative Methods of:

End-Quench Test for Hardenability of Steel (A 255 – 42 T), submitted by Committee A-1 on Steel,

Chemical Analysis of Soda-Lime Glass (C 169 – 41 T), submitted by Committee C-14 on Glass and Glass Products,

Test for Compressive Strength of Natural Building Stone (C 170 – 41 T), submitted by Committee C-18 on Natural Building Stones,

Conditioning of Paperboard, Fiberboard, and Paperboard Containers for Testing (D 641 – 41 T), submitted by Committee D-6 on Paper and Paper Products,

Compression Testing of Corrugated and Solid Fiber Boxes (D 642 – 41 T), submitted by Committee D-6,

Test for Folding Endurance of Paper (D 643 – 41 T), submitted by Committee D-6,

Test for Moisture in Paper and Paper Products (D 644 – 41 T), submitted by Committee D-6,

Test for Thickness of Paper (D 645 - 41 T), submitted by Committee D-6,
Test for Basis Weight of Paper and Paper Products (D 646 - 41 T), submitted by Committee D-6,
Test for Product Uniformity of Phenolic Laminated Sheets (D 634 - 41 T), submitted by Committee D-9 on Electrical Insulating Materials,
Testing Molded Materials Used for Electrical Insulation (D 48 - 41 T), submitted by Committee D-9,
Test for Distortion Under Heat of Molded Electrical Insulating Materials (D 648 - 41 T), submitted by Committee D-9,
Test for Compressive Strength of Electrical Insulating Materials (D 649 - 41 T), submitted by Committee D-9,
Test for Flexural Strength of Electrical Insulating Materials (D 650 - 41 T), submitted by Committee D-9,
Test for Tensile Strength of Electrical Insulating Materials (D 651 - 41 T), submitted by Committee D-9,
Test for Impact Resistance of Plastics and Electrical Insulating Materials (D 256 - 41 T), submitted jointly by Committee D-9 and Committee D-20 on Plastics,
Measuring Mica Stampings Used in Electronic Devices and Incandescent Lamps (D 652 - 41 T), submitted by Committee D-9,
Testing Asphalt Composition Battery Containers (D 639 - 41 T), submitted by Committee D-11 on Rubber Products,
Sampling and Testing Rubber Latex (D 640 - 41 T), submitted by Committee D-11,
Test for Flammability of Plastics Over 0.050 in. in Thickness (D 635 - 41 T), submitted by Committee D-20 on Plastics,
Test for Diffusion of Light by Plastics (D 636 - 41 T), submitted by Committee D-20,
Test for Surface Irregularities of Flat Transparent Plastic Sheet (D 637 - 41 T), submitted by Committee D-20,
Tension Testing of Plastics (D 638 - 41 T), submitted by Committee D-20,
Chemical Analysis of Aluminum, Columbium, and Lead in Steel (E 30 - 41 T), submitted by Committee E-3 on Chemical Analysis of Metals,

Tentative Definitions of:

Terms Relating to Lime (C 51 - 41 T), submitted by Committee C-7 on Lime,
Terms and Symbols Relating to Soil Mechanics (D 653 - 42 T), submitted by Committee D-18 on Soils for Engineering Purposes, and
Volume Correction Table for Tar and Coal-Tar Pitch (D 633 - 41 T), submitted jointly by Committee D-4 on Road and Paving Materials and Committee D-8 on Bituminous Waterproofing and Roofing Materials.

Revisions of the following tentative standards and tentative revisions of standards were accepted by Committee E-10:

Tentative Specifications for:

Iron and Steel Arc-Welding Electrodes (A 233 - 42 T), submitted by Committee A-1 on Steel,
Electrolytic Cathode Copper (B 115 - 41 T), submitted by Committee B-2 on Non-Ferrous Metals and Alloys,
Nickel-Copper Alloy Plate, Sheet, and Strip (B 127 - 41 T), submitted by Committee B-2,
Beryllium-Copper Alloy Rods, Bars, Sheet, Strip, and Wire (B 120 - 41 T), submitted by Committee B-5 on Copper and Copper Alloys, Cast and Wrought,
Building Brick (Made from Clay or Shale) (C 62 - 41 T), submitted by Committee C-15 on Manufactured Masonry Units,
Crushed Stone for Bituminous Macadam Base and Surface Courses (D 192 - 41 T), submitted by Committee D-4 on Road and Paving Materials,
Standard Sizes of Coarse Aggregate for Highway Construction (D 448 - 41 T), submitted by Committee D-4,
Crushed Slag for Bituminous Macadam Base and Surface Courses (D 487 - 41 T), submitted by Committee D-4,
A.S.T.M. Thermometers (E 1 - 41 T), submitted by Committee E-1 on Methods of Testing,

Tentative Methods of:

Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 88 - 41 T), submitted by Committee C-9 on Concrete and Concrete Aggregates,
Sampling Bituminous Materials (D 140 - 41 T), submitted by Committee D-4 on Road and Paving Materials,
Sampling and Testing Untreated Paper Used in Electrical Insulation (D 202 - 41 T), submitted by Committee D-9 on Electrical Insulating Materials,
Test for Thickness of Solid Electrical Insulation (D 374 - 41 T), submitted by Committee D-9,

Test for Power Factor and Dielectric Constant of Natural Mica (D 351 – 42 T), submitted by Committee D-9,

Chemical Analysis of Rubber Products (D 297 – 41 T), submitted by Committee D-11 on Rubber Products,

Test for Resistance of Plastics to Chemical Reagents (D 543 – 41 T), submitted by Committee D-20 on Plastics,

Impact Testing of Metallic Materials (E 23 – 41 T), submitted by Committee E-1 on Methods of Testing,

Standard Specifications for:

Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (B 4 – 27), submitted by Committee B-2 on Non-Ferrous Metals and Alloys,

Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (B 5 – 27), submitted by Committee B-2,

Phosphor Bronze Sheet and Strip (B 103 – 40), submitted by Committee B-5 on Copper and Copper Alloys, Cast and Wrought,

Clay Sewer Pipe (C 13 – 40), submitted by Committee C-4 on Clay Pipe,

Lightweight Aggregates for Concrete (C 130 – 39), submitted by Committee C-9 on Concrete and Concrete Aggregates,

A.S.T.M. Thermometers (E 1 – 39), submitted by Committee E-1 on Methods of Testing,

Standard Methods of:

Making and Storing Compression Test Specimens of Concrete in the Field (C 31 – 39), submitted by Committee C-9 on Concrete and Concrete Aggregates,

Test for Compressive Strength of Concrete (C 39 – 39), submitted by Committee C-9,

Securing Specimens of Hardened Concrete from the Structure (C 42 – 39), submitted by Committee C-9,

Test for Compressive Strength of Concrete Using Portions of Beams Broken in Flexure (Modified Cube Method) (C 116 – 39), submitted by Committee C-9,

Test for Specific Gravity and Absorption of Fine Aggregate (C 128 – 39), submitted by Committee C-9,

Test for Specific Gravity and Absorption of Coarse Aggregate (C 127 – 39), submitted by Committee C-9,

Test for Ductility of Bituminous Materials (D 113 – 39), submitted by Committee D-4 on Road and Paving Materials,

Chemical Analysis of Calcium Chloride (D 345 – 34), submitted by Committee D-4, and

Testing Shellac Used for Electrical Insulation (D 411 – 40), submitted by Committee D-9 on Electrical Insulating Materials.

ACTIONS UNDER EMERGENCY PROCEDURE

As indicated above, quite a few standing committees submitted recommendations to Committee E-10 under the Society's emergency procedure. These are listed below under emergency alternate provisions, emergency alternate specifications, and emergency standards. The emergency alternate provisions are issued in the form of stickers printed on pink paper for attachment to the standards in question. Each one bears a prefatory note reading as follows:

> These Emergency Alternate Provisions are issued by the American Society for Testing Materials in accordance with a special procedure in the interest of expediting procurement or conservation of materials during the period of National Emergency. They are intended for use where they may be considered by the purchaser of the material as a permissible alternate for the specific application or use desired.

The complete alternate specifications and new emergency standards are similarly published on pink stock to indicate their emergency nature and have a similar prefatory note.

EMERGENCY ALTERNATE PROVISIONS

Thus far fifty emergency alternate provisions have been accepted by Committee E-10 as follows:

Designation	Emergency Alternate Provisions in	Committee
	Standard Specifications for:	
EA – A 1	Open-Hearth Carbon-Steel Rails (A 1 – 39)	A-1
EA – A 26	Steel Tires (A 26 – 39)	A-1
EA – A 27	Carbon-Steel Castings for Miscellaneous Industrial Uses (A 27 – 39)	A-1
EA – A 67	Steel Tie Plates (A 67 – 33)	A-1
EA – A 87	Carbon-Steel and Alloy-Steel Castings for Railroads (A 87 – 36)	A-1
EA – A 148	Alloy-Steel Castings for Structural tural Purposes (A 148 – 36)	A-1
EA – A 160	Axle-Steel Bars for Concrete Reinforcement (A 160 – 39)	A-1
EA – A 194	Carbon and Alloy-Steel Nuts for Bolts for High-Pressure and High-Temperature Service to 1100 F. (A 194 – 40)	A-1

Designation	Emergency Alternate Provisions in	Committee
EA - A 215	Carbon-Steel Castings Suitable for Fusion Welding for Miscellaneous Industrial Uses (A 215 - 41)	A-1
EA - A 227	Hard-Drawn Steel Spring Wire (A 227 - 41)	A-1
EA - A 229	Oil-Tempered Steel Spring Wire (A 229 - 41)	A-1
EA - A 230	Carbon-Steel Valve Spring Quality Wire	A-1
EA - A 241	Hot-Worked High-Carbon Steel Tie Plates (A 241 - 41)	A-1
EA - A 167	Corrosion-Resisting Chromium-Nickel Steel Sheet, Strip, and Plate (A 167 - 41)	A-10
EA - A 177	High-Strength Corrosion-Resisting Chromium-Nickel Steel Sheet and Strip (A 177 - 39)	A-10
EA - A 240	Corrosion-Resisting Chromium-Nickel Steel Sheet, Strip, and Plate for Fusion-Welded Unfired Pressure Vessels (A 240 - 41)	A-10
EA - B 23	White Metal Bearing Alloys (Known Commercially as "Babbitt Metal") (B 23 - 26)	B-2
EA - B 60	Castings of the Alloy: Copper 88 per cent, Tin 8 per cent, Zinc 4 per cent (B 60 - 41)	B-5
EA - B 62	Composition Brass or Ounce Metal Castings (B 62 - 41)	B-5
EA - D 82	Basic Sulfate White Lead (D 82 - 41)	D-1
EA - D 27	Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound (D 27 - 41)	D-11
EA - D 353	Insulated Wire and Cable: Performance Rubber Compound (D 353 - 41)	D-11
EA - D 469	Insulated Wire and Cable: Heat-Resisting Rubber Compound (D 469 - 41)	D-11
EA - D 455	Milled Toilet Soap (D 455 - 39)	D-12
EA - D 496	Chip Soap (D 496 - 39)	D-12
EA - D 498	Powdered Soap (Nonalkaline Soap Powder) (D 498 - 39)	D-12
EA - D 499	White Floating Toilet Soap (D 499 - 39)	D-12
EA - D 533	Built Soap, Powdered (D 533 - 41)	D-12
EA - D 535	Palm Oil Solid Soap (Type A, Pure; Type B, Blended) (D 535 - 41)	D-12
EA - A 183	Heat-Treated Carbon- and Alloy-Steel Track Bolts and Nuts (A 183 - 40 T)	A-1
EA - A 216	Carbon-Steel Castings Suitable for Fusion Welding for Service at Temperatures up to 850 F. (A 216 - 41 T)	A-1
EA - A 217	Alloy-Steel Castings Suitable for Fusion Welding for Service at Temperatures from 750 to 1100 F. (A 217 - 41 T)	A-1
EA - B 158	Rope-Lay-Stranded and Bunch-Stranded Copper Cables for Electrical Conductors (B 158 - 41 T)	B-1
EA - B 32	Soft Solder Metal (B 32 - 40 T)	B-2
EA - B 30	Copper-Base Alloys in Ingot Form for Sand Castings (B 30 - 41 T)	B-5
EA - B 143	Tin-Bronze and Leaded Tin-Bronze Sand Castings (B 143 - 41 T)	B-5
EA - B 144	High-Leaded Tin-Bronze Sand Castings (B 144 - 41 T)	B-5
EA - B 145	Leaded Red Brass and Leaded Semi Red Brass Sand Castings (B 145 - 41 T)	B-5
EA - B 146	Leaded Yellow Brass Sand Castings for General Purposes (B 146 - 41 T)	B-5
EA - B 148	Aluminum-Bronze Sand Castings (B 148 - 41 T)	B-5
EA - B 85	Aluminum-Base Alloy Die Castings (B 85 - 39 T)	B-6
EA - B 86	Zinc-Base Alloy Die Castings (B 86 - 41 T)	B-6
EA - D 224	Asphalt Roofing Surfaced with Powdered Talc or Mica (D 224 - 41 T)	D-8
EA - D 249	Asphalt Roofing Surfaced with Coarse Mineral Granules (D 249 - 41 T)	D-8
EA - D 532	Rubber Sheath Compound for Electrical Insulated Cords and Cables (D 532 - 39 T)	D-11
EA - D 574	Insulated Wire and Cable: Ozone-Resistant Type Insulation (D 574 - 40 T)	D-11
EA - D 536	Palm Oil Chip Soap (Type A, Pure; Type B, Blended) (D 536 - 41 T)	D-12
EA - D 592	Olive Oil Solid Soap (Type A, Pure; Type B, Blended) (D 592 - 41 T)	D-12
EA - D 593	Salt-Water Soap (D 593 - 40 T)	D-12
EA - D 630	Olive Oil Chip Soap (Type A, Pure; Type B, Blended) (D 630 - 41 T)	D-12

EMERGENCY ALTERNATE SPECIFICATIONS

One complete emergency alternate standard has been accepted by Committee E-10, namely, Emergency Alternate Specifications for Portland Cement (EA - C 150), submitted by Committee C-1.

EMERGENCY STANDARDS

Six emergency standards have been accepted by Committee E-10 as follows:

Emergency Specifications for:

Carbon-Chromium Ball and Roller Bearing Steels (ES - 5),

Hot-Dip Lead Coating on Iron and Steel Hardware (ES - 2), submitted by Committee A-5 on Corrosion of Iron and Steel,

Lead-Coated and Lead-Alloy-Coated Copper Wire for Electrical Purposes (ES - 1), submitted by Committee B-1 on Copper and Copper Alloy Wires for Electrical Conductors,

Rubber Sheath Compound for Electrical Insulated Cords and Cables Where Extreme Abrasion Resistance is not Required (ES - 6), submitted by Committee D-11 on Rubber Products,

Emergency Method for Conducting Salt Spray Tests on Organic Protective Coatings (ES - 3), submitted by Committee D-1 on Paint, Varnish, Lacquer, and Related Products, and

Emergency Standard Hardness Conversion Table for Cartridge Brass (ES - 4), submitted by Committee E-1 on Methods of Testing.

The Emergency Specifications for Carbon-Chromium Ball and Roller Bearing Steels (ES - 5) were referred directly to Committee E-10 by the Executive Committee of the Society upon their preparation by the Bearing Steel Section functioning under the NESS project of the War Production Board.

This section, under the chairmanship of L. A. Lanning, New Departure Division, General Motors Corp., is a subcommittee of the important NESS Technical Advisory Committee on Carbon and Alloy Steel Bars, Blooms, and Billets, and includes in its personnel leading metallurgists and technical men representing industrial consumers and general interests from the roller bearing and ball bearing fields, and steel producers. It was organized basically along the same lines as an A.S.T.M. group, that is, of producers, consumers, and general interests, and the S.A.E. and the A.I.S.I., co-sponsors with A.S.T.M. of the NESS project, agreed that the A.S.T.M. was the logical one of the sponsors to promulgate the specifications. They were submitted to Committee E-10 on the basis of the manner in which they had been developed and the considerations that had entered into their development, and subsequent to approval by Committee E-10, the specifications were referred to Committee A-1 on Steel in case that committee should wish to include specifications for this material in its standardization program. The latter committee will discuss the specifications during the annual meeting and if a decision is reached to sponsor similar A.S.T.M. standards, a subcommittee will undoubtedly be appointed to be responsible for such specifications.

Promotion and Expansion of Standardization Activities

As might well be expected in view of the activities in other directions, very little thought has been given to expansion of the Society's standardization activities along new lines. The committee has felt that the time is not particularly opportune for undertaking any new work in view of the pressure under which most individuals are now working in connection with war activities. The committee still has before it several proposals that have been made from time to time as for example the work on adhesives and waxes and consideration will be given to these topics at the appropriate time.

Relations with the American Standards Association

Standards Submitted Under Existing Standards and Proprietary Procedure.—The existing standards method for submitting standards to the American Standards Association whereby it is necessary to show that the standard is supported by the necessary consensus of those substantially concerned with its scope and provisions, is particularly appropriate for a large number of A.S.T.M. standards. Under this procedure, the following specifications and methods have been approved during the year by the ASA as American standard:

Standard Specifications for:

Calcined Gypsum for Dental Plasters (C 72 – 40; ASA A65.1–1941),
Keene's Cement (C 61 – 40; A66.1–1941),
Gypsum Lath (C 37 – 40; A67.1–1941),
Gypsum Sheathing Board (C 79 – 34; A68.1–1941),
Gypsum Wall Board (C 36 – 34; A69.1–1941),
Concrete Masonry Units for Construction of Catch Basins and Manholes (C 139 – 39; A73.1–1942),
Structural Clay Load-Bearing Wall Tile (C 34 – 41; A74.1–1942),
Concrete Building Brick (C 55 – 37; A75.1–1942),
Structural Clay Non-Load-Bearing Tile (C 56 – 41; A76.1–1942),
Structural Clay Floor Tile (C 57 – 39; A77.1–1942),
Sand-Lime Building Brick (C 73 – 39; A78.1–1942),
Hollow Load-Bearing Concrete Masonry Units (C 90 – 39; A79.1–1942),
Hollow Non-Load-Bearing Concrete Masonry Units (C 129 – 39; A80.1–1942),
Solid Load-Bearing Concrete Masonry Units (C 145 – 40; A81.1–1942),
Gray Iron Castings (A 48 – 41; G25.1–1942),

Cast-Iron Culvert Pipe (A 142 - 38; G26.1-1942),
Lightweight and Thin-Sectioned Gray-Iron Castings (A 190 - 40; G27.1-1942),
Boiler Rivet Steel and Rivets (A 31 - 40; G28.1-1942),
Carbon-Steel Plates for Stationary Boilers and Other Pressure Vessels (A 70 - 39; G29.1-1942),
Low Tensile Strength Carbon-Steel Plates of Flange and Firebox Qualities (A 89 - 39; G30.1-1942),
Carbon-Silicon Steel Plates of Ordinary Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels (A 201 - 39; G31.1-1942),
Chrome-Manganese-Silicon (CMS) Alloy-Steel Plates for Boilers and Other Pressure Vessels (A 202 - 39; G32.1-1942),
Low-Carbon Nickel-Steel Plates for Boilers and Other Pressure Vessels (A 203 - 39; G33.1-1942),
Molybdenum-Steel Plates for Boilers and Other Pressure Vessels (A 204 - 39; G34.1-1942),
High-Tensile Strength Carbon-Silicon Steel Plates for Boilers and Other Pressure Vessels (Plates 4½ in. and Under in Thickness) (A 212 - 39; G35.1-1942),
Alloy-Steel Castings for Valves, Flanges, and Fittings for Service at Temperatures from 750 to 1100 F. (A 157 - 41; G36.1-1942),
Forged or Rolled Alloy-Steel Pipe Flanges, Forged Fittings, and Valves and Parts for Service at Temperatures from 750 to 1100 F. (A 182 - 40; G37.1-1942),
Carbon and Alloy-Steel Nuts for Bolts for High-Pressure and High-Temperature Service to 1100 F. (A 194 - 40; G38.1-1942),
Structural Steel for Locomotives and Cars (A 113 - 39; G39.1-1942),
Low Tensile Strength Carbon-Steel Plates of Structural Quality for Welding (A 78 - 40; G40.1-1942),
Structural Silicon Steel (A 94 - 39; G41.1-1942),
High-Strength Structural Rivet Steel (A 195 - 41; G42.1-1942),
Axle-Steel Bars for Concrete Reinforcement (A 160 - 39; G43.1-1942),
Fabricated Steel Bar or Rod Mats for Concrete Reinforcement (A 184 - 37; G44.1-1942),
Welded Steel Wire Fabric for Concrete Reinforcement (A 185 - 37; G45.1-1942),
Malleable Iron Castings (A 47 - 33; G48.1-1942),
Cupola Malleable Iron (A 197 - 39; G49.1-1942),
Basic Sulfate White Lead (D 82 - 41; K47-1941),
Blue Lead; Basic Sulfate (D 405 - 41; K48-1941),
C.P. Para Red Toner (D 475 - 41; K49-1941),
C.P. Zinc Yellow (Zinc Chromate) (D 478 - 41; K50-1941),
Textile Testing Machines (D 76 - 41; L15.1-1942),

Standard Methods of:

Testing Gypsum and Gypsum Products (C 26 - 40; A70.1-1941),
Sampling and Testing Brick (C 67 - 41; A82.1-1942),
Sampling and Testing Structural Clay Tile (C 112 - 36; A83.1-1942),
Sampling and Testing Concrete Masonry Units (C 140 - 39; A84.1-1942),
Sample Preparation for Physical Testing of Rubber Products (D 15 - 41; J1.1-1942),
Tension Testing of Vulcanized Rubber (D 412 - 41; J2.1-1942),
Test for Adhesion of Vulcanized Rubber (Friction Test) (D 413 - 39; J3.1-1942),
Test for Accelerated Aging of Vulcanized Rubber by the Oxygen-Pressure Method (D 572 - 41; J4.1-1942),
Test for Accelerated Aging of Vulcanized Rubber by the Oven Method (D 573 - 41; J5.1-1942),
Test for Alkalinity or Acidity of Pigments (D 278 - 31; K51-1941),
Test for Bleeding of Pigments (D 279 - 31; K52-1941),
Test for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments (D 280 - 33; K53-1941),
Test for Oil Absorption in Pigments (D 281 - 31; K54-1941),
Test for Acetone Extract in Dry Lampblack and Dry Bone Black (D 305 - 31; K55-1941),
Test for Tinting Strength of White Pigments (D 332 - 36; K56-1941),
Test for Mass Color and Tinting Strength of Color Pigments (D 387 - 36; K57-1941),
Chemical Analysis of Yellow and Orange Pigments Containing Chromium Compounds, Blue Pigments, and Chrome Green (D 126 - 36; K58-1941),
Chemical Analysis of Dry Mercuric Oxide (D 284 - 33; K59-1941), and
Testing Wool Felt (D 461 - 40; L16.1-1942).

The Society has been granted proprietary sponsorship for the revisions of standards approved under the existing standards method and, functioning as proprietary sponsor, has submitted to the ASA during the year revisions of the following standards, all of which have been approved:

Standard Specifications for:

Gypsum (C 22 - 41; ASA A49.1-1941),
Carbon-Steel Castings for Valves, Flanges, and Fittings for High-Temperature Service (A 95 - 41; G17.1-1942),
Zinc Oxide (D 79 - 41; K22-1941),
Basic Carbonate White Lead (D 81 - 41; K23-1941),
Red Lead (D 83 - 41; K24-1941),
Mineral Iron Oxide (D 84 - 41; K25-1941),
Lampblack (D 209 - 41; K26-1941),
Chrome Yellow (D 211 - 41; K27-1941),
Reduced Chrome Green (D 213 - 41; K28-1941),
Prussian Blue (D 261 - 41; K29-1941),
Reduced Para Red (D 264 - 41; K31-1941),
Bone Black (D 210 - 41; K36-1941),
Chrome Oxide Green (D 263 - 41; K37-1941),
Titanium Dioxide Pigments (D 476 - 41; K45-1941), and

Standard Methods of:

Testing and Tolerances for Tubular Sleeving and Braids (D 354 - 41; L13.1-1942).

Standards Submitted Under Sectional Committee Procedure.—Reports were received from several of the sectional committees for which the Society is sponsor, submitting standards to the ASA for approval. These reports were transmitted to that association and the following standards have been approved for the first time or reapproved in revised form during the year as American standard:

On the Recommendation of Sectional Committee B36 on Standardization of Dimensions and Materials of Wrought-Iron and Wrought-Steel Pipe and Tubing:

Specifications for Lap-Welded and Seamless Steel Pipe for High-Temperature Service (A 106 - 41; ASA B36.3-1942).

On the Recommendation of Sectional Committee C59 on Electrical Insulating Materials:

Specifications for Electrical Gloves (Voltage Rating of Gloves - 3000 Volts) (D 120 - 40; C59.12-1942).

On the Recommendation of Sectional Committee H4 on Copper Wire:

Specifications for Soft or Annealed Copper Wire (B 3 - 41; H4.1-1942), and
Specifications for Hot-Rolled Copper Rods for Electrical Purposes (B 49 - 41; H4.7-1942).

On the Recommendation of Sectional Committee Z11 on Petroleum Products and Lubricants:

Method of Test for Carbon Residue of Petroleum Products (Conradson Carbon Residue) (D 189 - 41; Z11.25-1941),
Method of Test for Distillation of Gas Oil and Similar Distillate Fuel Oils (D 158 - 41; Z11.26-1941),
Method of Test for Distillation of Crude Petroleum (D 285 - 41; Z11.32-1941),
Method of Test for Distillation of Plant Spray Oils (D 447 - 41; Z11.43-1941),
Method of Test for Vapor Pressure of Petroleum Products (Reid Method) (D 323 - 41; Z11.44-1941),
Method for Calculating Viscosity Index (D 567 - 41; Z11.45-1941), and
Method for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity (D 446 - 39; Z11.46-1941).

On the recommendation of Sectional Committee A1 on Specifications for Portland Cement, the approval as American standard of the following obsolete standards was withdrawn by the ASA:

Specifications for Portland Cement (C 9 - 30; A1a-1931), and
Methods of Testing Cement (C 77 - 32; A1.2-1933).

On the recommendation of Sectional Committee Z11 on Petroleum Products and Lubricants, the approval as American standard of the following standard, which has been revised and reverted to tentative, has been withdrawn by the ASA:

Method of Test for Knock Characteristics of Motor Fuels (D 357 - 40; Z11.37-1940).

SECTIONAL COMMITTEES ORGANIZED

In order to coordinate the work on colorfastness, a new Sectional Committee L14 on Fastness of Colored Textiles was organized during the past year under the procedure of the American Standards Association. The Society is serving with the American Association of Textile Chemists and Colorists as joint sponsor of this committee and is represented thereon by A. G. Ashcroft and W. M. Scott with R. E. Hess as alternate.

A new Sectional Committee A51 on Building Code Requirements for Fire Protection and Fire Resistance, recently organized under the procedure of the American Standards Association, is also of interest to the A.S.T.M. The Society has accordingly appointed H. M. Robinson as representative and G. N. Thompson as alternate thereon.

The reorganization of Sectional Committee A37 on Road and Paving Materials, mentioned in the 1941 report, has now been completed. The Society is sponsor of this committee and is represented thereon by E. F. Kelley, F. V. Reagel, and C. S. Reeve.

Respectfully submitted on behalf of the committee,

J. R. TOWNSEND,
Chairman.

C. L. WARWICK,
Ex-officio Secretary.

TECHNICAL PAPERS

With Discussions

EDGAR MARBURG LECTURE

THE PURPOSE of the Edgar Marburg Lecture is to have described at the annual meetings of the American Society for Testing Materials, by leaders in their respective fields, outstanding developments in the promotion of knowledge of engineering materials. Established as a means of emphasizing the importance of promoting knowledge of materials, the Lecture honors and perpetuates the memory of Edgar Marburg, first secretary of the Society.

GASOLINE—PAST, PRESENT AND FUTURE

SEVENTEENTH EDGAR MARBURG LECTURE[1]

By Graham Edgar[2]

FOREWORD

The American Society for Testing Materials defines gasoline as "a refined petroleum naphtha which by its composition is suitable for use as a carburant in internal combustion engines."[3] "Petroleum Naphtha" is defined as "a generic term applied to refined, partly refined or unrefined petroleum products and liquid products of natural gas not less than 10 per cent of which distills below 347 F. (175 C.) and not less than 95 per cent of which distills below 464 F. (240 C.), when subjected to distillation in accordance with the Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (A.S.T.M. Designation: D 86)."

Broad definitions are difficult to draw and easy to find fault with. For the purpose of the following discussion, it is necessary to limit the field by inserting the words "spark ignition" before "internal combustion engines" in the definition of gasoline.

The most important words in the definition are these: "suitable for use . . . in internal combustion engines." For the properties of gasoline are inseparably linked with the properties of the engine for which it is to be used. There are no ideal properties of gasoline, except in so far as particular gasoline engines are concerned; and there are no ideal properties of internal combustion engines except as related to the fuel which they are to use. The characteristics of the engine must be adapted to the characteristics of available gasoline, and the characteristics of gasoline must be modified to meet the needs of engines. The history of engines and fuels is filled with such adaptations and modifications. Therefore, any consideration of gasoline must involve the simultaneous consideration of engines, and in the following discussion this theme will recur a number of times.

THE SPARK IGNITION INTERNAL COMBUSTION ENGINE

In its simplest form, that of a single-cylinder engine, the spark ignition internal combustion engine is a heat engine, igniting a combustible mixture, and converting a portion of the heat into useful power. Gasoline for such an engine is "suitable" if it is sufficiently volatile to form an explosive mixture with air, and if its nature is such as to permit the mixture to burn "normally" in the engine, that is, without preignition or knock. The earliest engines were of this type, and a wide range of fuels was suitable for them. The gradual changes which have led to the multicylinder automobile engine of today and the aircraft engine of today have imposed increasing restrictions upon those qualities which determine the suitability of gasoline for the engines in question, and have led to the necessity of the development of a considerable number of tests designed to determine those properties of gasoline which make it "suitable" for the engine in which it is to be employed. The development and perfection of such tests is, of course, the province of the American Society for Testing Materials, although in the case of gasoline, the A.S.T.M. and the Cooperative Fuel Research Committee have cooperated in many matters.

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[1] Read on June 24, 1942, before the Annual Meeting of the American Society for Testing Materials, Atlantic City, N. J.

[2] Vice-President, and Director of Research, Ethyl Corp., New York, N. Y.

[3] Standard Definitions of Terms Relating to Petroleum (D 288-39), 1939 Book of A.S.T.M. Standards, Part III, p. 236.

THE A.S.T.M. TESTS FOR GASOLINE

The A.S.T.M. has, so far, employed the following tests for gasoline:[4]

(1) Acid heat (D 481 – 39)
(2) Color (D 156 – 38)
(3) Distillation (D 86 – 40 and D 216 – 40)
(4) Gravity (D 287 – 39)
(5) Gum content (D 381 – 36)
(6) Gum stability (D 525 – 41T)
(7) Knock characteristics (D 357 – 41T) and (D 614 – 41T)
(8) Sulfur (free and corrosive) (D 130 – 30)
(9) Sulfur (total) (D 90 – 34 T)
(10) Tetraethyl lead (D 526 – 41 T)
(11) Vapor pressure (D 323 – 41)
(12) Flash point (D 56 – 36)
(13) Viscosity (D 446 – 39) and (D 445 – 39 T)

It appears desirable to consider briefly the significance of some of these tests.

Of these tests, those for acid heat, color, and gravity are of importance only for identification purposes and do not in themselves determine the suitability of gasoline for use in an internal combustion engine. There appears to be no reason to consider them in detail.

The tests for gum content, gum stability, free and corrosive sulfur, and total sulfur are designed to detect and determine the existence and amount of "impurities" (or in one case, the tendency to form such impurities in storage) which might affect adversely the operations of the internal combustion engine.

"Gum" is the term applied to the sticky or shellac-like residue which may be formed when a sample of gasoline is evaporated. Excessive amounts of it will cause sticking of valves and clogging of carburetor jets, and the gum content of gasoline must, therefore, be kept within narrow limits. The A.S.T.M. test for gum content (a rapid evaporation in a current of air, the residue being weighed) is believed to give results which will predict reasonably well the suitability of the gasoline for an automotive engine, as far as difficulties of operation are concerned. "Gum stability," however, is quite another matter. Freshly distilled gasoline contains no gum but may contain unstable (usually olefinic or diolefinic) hydrocarbons, which through oxidation and polymerization will form gum. The A.S.T.M. test for gum stability (essentially an accelerated storage test, in which the sample is heated at 100 C. under 100 lb. oxygen pressure, measurements being made of the drop in pressure with time, and of the gum formed by the end of the test) is an attempt to predict the rate and amount of gum formation in the gasoline under storage conditions. It cannot be said that the results of this test are entirely satisfactory. They distinguish a relatively stable gasoline from a relatively unstable one, but they cannot be used to estimate with any accuracy the length of time in which a given gasoline may be stored without formation of excessive gum, unless correlated with actual storage data on gasoline of similar type. For this reason, the refiner usually "plays safe" and adds to his gasoline gum inhibitors (usually oxidation inhibitors) in amounts more than adequate to provide reasonable storage life. It may be noted that the A.S.T.M. has so far not adopted any tests to determine the presence, nature, or amounts of gum inhibitors in gasoline. Perhaps steps should be taken in this direction.

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[4] 1939 Book of A.S.T.M. Standards, and the 1940 and 1941 Supplements, Part III.

Free or corrosive sulfur in gasoline is objectionable but easily controlled. Total sulfur (determined by burning a sample of gasoline in a lamp, and absorbing and determining the sulfuric acid thus formed) furnishes another illustration of the close relationship between gasoline properties and automotive engine design. In the early days of the automobile, instances occurred of corroded crankshafts, wrist pins, etc., in gasolines of higher than average sulfur content. This was attributed to condensation of water in the crankcase during cold weather operation, and absorption of sulfuric acid in this water. As a result, gasoline specifications invariably limited sulfur to 0.1 per cent, a limit which, in many cases, worked an economic hardship on the petroleum industry. With the introduction by the automotive industry some years ago of crankcase ventilation, minimizing condensation of water, corrosion complaints due to sulfur in gasoline have practically disappeared, and the specification limits on sulfur have, in many cases, been either raised or abandoned. In fact, it seems today doubtful whether there is any reason for limiting the total sulfur in gasoline.

The test for tetraethyl lead in gasoline is to determine the presence and amount of an additive commonly used to increase antiknock value.

The tests having the greatest bearing on the suitability of gasoline for use in internal combustion engines are those relating to volatility (distillation, vapor pressure) and those relating to knocking tendency. These deserve somewhat detailed consideration.

Distillation:

This test for gasoline involves a simple (nonfractionating) distillation of a 100-ml. sample in a glass flask equipped with thermometer and condenser. The data recorded are:

(*a*) The temperature at which the first drop of distillate is observed (initial boiling point); (*b*) the volume of distillate condensed at oven temperature intervals of 10 C.; (*c*) the maximum temperature observed; (*d*) the total volume of distillate recovered; (*e*) the undistilled residue; (*f*) the difference between 100 ml. and the sum of (*c*) and (*e*), recorded as "distillation loss."

There appears, at first sight, to be no reason to expect that such a simple distillation test would give any accurate information on the vaporization characteristics of gasoline in an engine where the evaporation of the fuel is brought about in a current of air. However, many careful comparisons of A.S.T.M. distillation data with those involving evaporation in an air stream, and with actual car performance, have shown that nearly all important volatility characteristics of gasoline can be calculated from the simple distillation data.

From these studies it has been found, for example, that the 10 per cent point, particularly in combination with the slope of the temperature – volume recovered graph, is related to the ease with which an engine may be started, particularly in cold weather; the lower the 10 per cent point, the lower the temperature at which satisfactory starting will occur. The 90 per cent point is a good measure of the completeness with which gasoline may be expected to evaporate when used in the automotive engine; and hence as a measure of possible dilution of the lubricating oil (crankcase dilution). The higher the 90 per cent point, the greater the tendency towards crankcase dilution. The 50 per cent point may be taken as a rough measure of the over-all average volatility of gasoline, relating to its tendency to evaporate sufficiently to give reasonable distribution among the cylinders at moderate speeds. Numerous efforts have been made to correlate other distillation data with the performance of the automobile engine under various operating conditions, but the three points noted above are the ones usually regarded as most important, and these are the only ones usually included in gasoline specifications.

It may be pointed out that the importance of the distillation characteristics of gasoline are largely determined by the fact that the automobile engine (and the aircraft engine) of today is a multicylinder engine in which the fuel air mixture is obtained by means of a carburetor. If the fuel were supplied to the individual cylinders by direct injection instead of by a carburetor (as has been done in some aircraft engines), the distillation characteristics of gasoline would be of much less importance, and the interpretation of the distillation data in terms of engine performance would be quite different—another illustration of the close relationship between the characteristics of the fuel and those of the engine.

Vapor Pressure:

The vapor pressure of gasoline is, like the 10 per cent point, related to ease of starting, but its greatest value is in evaluating (1) the tendency of gasoline to evaporate in storage or handling, and (2) the tendency to boil in the fuel system (in aircraft at high altitude, in the fuel tank as well) and cause interruption of gasoline flow to the engine (vapor lock). The higher the vapor pressure, the greater the tendency to evaporate or to boil.

We have here a good illustration of the fact that there are no ideal properties of gasoline. In general, the higher the vapor pressure, the lower the temperature at which an automobile will start easily, but also the lower the temperature of the fuel system must be to avoid vapor lock. The refiner, therefore, endeavors to market a satisfactory compromise, namely, a gasoline which will give easy starting at the lowest expected temperature and at the same time will not cause vapor lock at the highest expected temperature of the fuel system. This obviously means that the volatility characteristics of the gasoline must be adjusted both seasonally and geographically, and that they may be expected to change with changes made in the automobile fuel system.

In the very early days of gasoline, it was largely a waste product, kerosine being the product most in demand. As a result, the earliest gasolines were extremely volatile, as the refiner attempted to include in the kerosine fraction as low a boiling range as was possible without too low a flash point. With the tremendous demand for gasoline created by the automobile, however, it became necessary to "stretch" the gasoline fraction as far as possible, and as a result, gasoline became so involatile as to require the automobile engineer to equip his cars with "hot spots," or heat on the manifold in some form, in order to achieve reasonable distribution of the fuel-air mixture among the cylinders. However, with the development of "cracking," or the breaking down of the heavier constituents of petroleum into lighter ones, it became technically and economically possible to increase still further yields of gasoline and at the same time to make the volatility almost anything that might be desired. As a result, for the past fifteen years, there has been a steady increase in the volatility of gasoline, the heat on the manifold of the automobile has gradually disappeared, and the automotive engineer has had the opposite problem, namely, that of decreasing his fuel line temperatures and modifying his carburetors to avoid vapor lock.

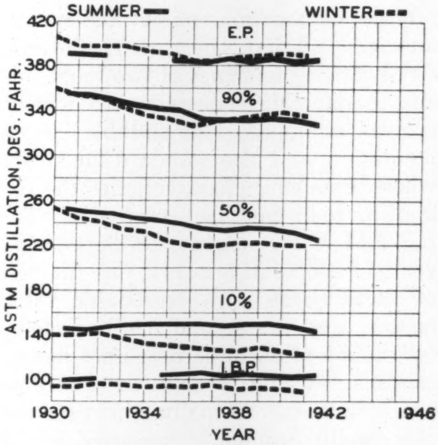

FIG. 1.—Trend in Volatility of Premium Grade Gasoline.

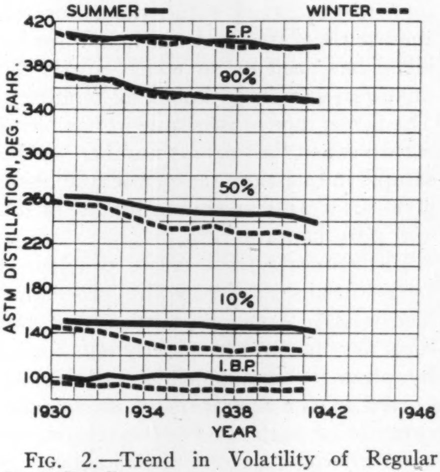

FIG. 2.—Trend in Volatility of Regular Grade Gasoline.

Figures 1, 2, 3, and 4 show the trends in average motor gasoline volatility for the past fifteen years. They show a steady increase in over-all volatility, which appears, however, to be stabilizing somewhat during the past few years.

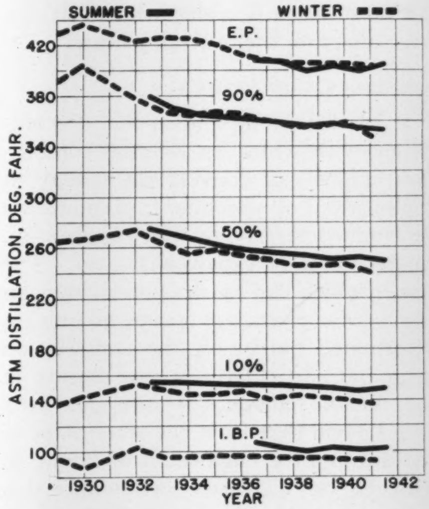

FIG. 3.—Trend in Volatility of Third Grade Gasoline.

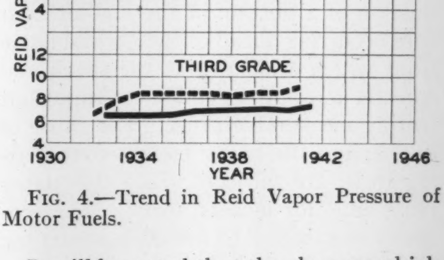

FIG. 4.—Trend in Reid Vapor Pressure of Motor Fuels.

It will be noted that the changes which have occurred in the volatility of gasoline have been very gradual changes. The very magnitude of the petroleum industry makes this inevitable. The improvements in refining processes and in refining technique are usually made in small increments. And, even if some quite revolutionary process appears, it is generally some years before its adoption can be sufficiently widespread to have a large affect on the over-all picture.

Knocking Characteristics:

It has been long known that the efficiency with which an internal combustion engine can convert the energy of combustion of gasoline into power is determined largely by the compression (more correctly expansion) ratio. The power which can be developed from an engine of a given size (swept volume) is determined partly by the compression ratio, but particularly by the density of the fuel-air charge. It has also been long known that from the standpoint of the automotive engineer, the extent to which he can increase either compression ratio or charge density (as by supercharging) is limited by the tendency shown in varying degrees by all fuels to undergo in the engine an abnormal type of combustion giving rise to power loss, and to such physical characteristics as high pitched sound waves and excessive temperatures. This abnormal combustion is generally known as "knock," and many elaborate investigations have shown that it is an extremely rapid combustion of a portion of the fuel-air mixture occurring ahead of the normal flame front which originates at the spark and spreads across the cylinder at rapid but readily measurable rates. The cause of knock, and the many factors contributing to it, are subjects which this paper must omit for lack of space. The tendency of gasoline to knock, is, therefore, perhaps the most important single property, as far as the ultimate development of the internal combustion engine is concerned.

The measurement of the knocking tendency of gasoline has been one of the most difficult problems confronting the A.S.T.M. It has been found impossible to measure this property satisfactorily by any method not utilizing an actual internal combustion engine; and even in an engine, all that is measured is the relative knocking tendency of two feuls. It has, therefore, been necessary to develop engines equipped with suitable instrumentation, and also standard fuels with whose knocking characteristics the unknown fuel can be compared. The A.S.T.M.-C.F.R. engine, developed after years of cooperative study, fills the first of these needs with reasonable satisfaction, and the second has been filled by the adoption of certain pure hydrocarbons varying widely in knocking characteristics. Actually, in practice, the gasoline sample is compared in the engine with various blends of n-heptane, and *iso*octane (2,2,4 trimethyl pentane) (or with secondary standards calibrated against these two hydrocarbons) until a blend has been found which equals the unknown sample in knocking tendency. The percentage of *iso*octane in this blend is then called the "octane number" of the sample in question, pure *iso*octane being given arbitrarily an octane number of 100 and pure heptane zero.

Unfortunately, the entire problem is by no means solved by this procedure. The relative knocking tendency of two fuels may be, and frequently is, affected by almost every possible variable involved in the operation of an internal combustion engine. For this reason, the octane number determined as above has significance only for the specific engine and for the specific set of operating conditions selected. It is, therefore, necessary to attempt to select conditions which will reflect, as closely as may be practicable, the behavior of gasoline in the automobile and aircraft engines for which they are to be used. This problem has already been the subject of years of experiment and debate, and is much too involved for a detailed discussion in the present paper. Out of the welter of experiment and statistical survey have come four sets of conditions for knock testing, two for motor gasolines and two for aviation gasolines. At the present time, the best technical opinion concerning these tests may be summarized as follows:

1. The standard A.S.T.M.-C.F.R. Method reflects fairly well the average road performance of conventional gasolines in the average automobile operating under rather severe conditions of load and speed.

2. The C.F.R. Research Method reflects fairly well the average road performance of conventional gasolines in the average automobile operating under rather light conditions of load and speed.

3. The difference in the octane number of a fuel by the two test methods is a fair measure of the knocking characteristics of the fuel to change with conditions of test ("sensitivity").

4. Neither method can be relied on to correspond accurately to average "customer acceptance" of the fuel, nor especially to predict the performance of any specific fuel in any specific automobile.

5. The octane number of motor gasoline has its most important value to the refiner in enabling him to check the performance of his refining processes and must be supplemented by careful and extensive road tests in automobiles before a correct evaluation of road performance can be obtained. It is perhaps to be regretted that laboratory octane numbers have found their way so extensively into the sales and advertising departments of the petroleum industry, where their limitations and interpretation may not be too well understood.

6. For aviation fuels the present 1C Method gives results which correlate reasonably well with the average full scale aircraft engine behavior under conditions of lean fuel-air mixtures, while the 3C Method correlates reasonably well with the full scale engine behavior under take-off conditions.

Figure 5 shows the trend in octane numbers (A.S.T.M.) of the different grades of gasoline during the past fifteen or more years. Here again the trends have been gradual, save for a fairly sudden rise in octane number in "regular grade" gasoline in 1933 to 1934, reflecting the addition of tetraethyl lead to this grade by a considerable proportion of the petroleum industry, and a similar rise in the octane number of third-grade gasoline about 1938 for the same reason.

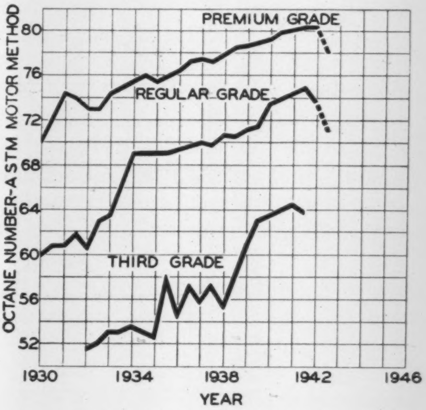

FIG. 5.—Trends in Antiknock Quality of Motor Fuels.

It is obvious from these charts that the limitations imposed upon the development of the internal combustion engine by the knocking tendency of gasoline have been gradually lifted over a period of many years. To what extent has the automotive engineer taken advantage of the opportunity thus offered to improve the efficiency of his engines? Figure 6 indicates that the automotive industry has been quick to take advantage of these trends in increasing antiknock value.

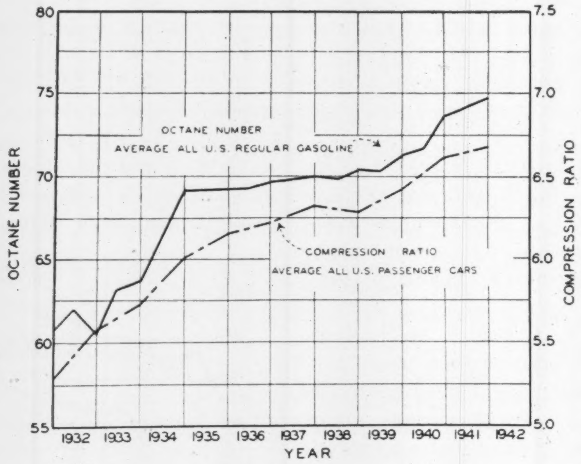

FIG. 6.

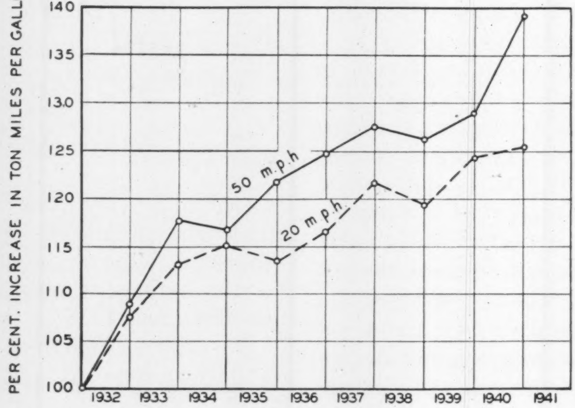

FIG. 7.

A graph of average compression ratios of passenger automobiles is roughly parallel to that of the average octane number of regular grade gasoline. This does not mean, of course, that all engines have had identical compression ratios or fuel antiknock requirements. Some engines have been designed for premium grade gasoline, and others have required its use, either through carbon accumulation, manufacturing variation, or purchaser adjustment. Others have been designed for lower than average antiknock quality. Furthermore, the time lag involved in the operations of both the automotive and petroleum industries hinders a completely parallel development. The automobile engine driven in one year will have been developed the year before, and will have been tested on fuels available during that year, and designed for such fuels, unless the designer chooses to gamble a bit on fuel improvement. As a result, there have been times when the average fuel has been of higher octane number than the average engine required, but there have also been times when the automotive engineers' estimates have been optimistic, and many engines at least have required better than average fuels. The net result of this "competition," however, has been a pretty close correlation between engine requirements and fuel quality, and the public has benefited therefrom, as illustrated by the data in Fig. 7, in which it is shown that ton-miles per gallon have increased steadily as fuel quality has increased. It would be an exaggeration to attribute all of this increase to increases in fuel antiknock value, but this has been a most important factor.

TENDENCY TO PREIGNITION

In addition to knock, another type of abnormal combustion is preignition, and this has become of increasing importance in the past few years. Preignition is, in general, an ignition of the fuel-air mixture by some local hot surface in the combustion chamber, perhaps an inadequately cooled area, or perhaps a deposit of "carbon." The phenomenon is much more erratic than knock and much more difficult to measure. The A.S.T.M. has so far made no attempt to devise and standardize methods of measuring the preignition tendencies of gasoline, but as it is most prevalent in high-compression engines, and thus like knocking tendency limits the development of the automotive engine, it would appear desirable that suitable technique for its measurement should be developed.

THE CHEMISTRY OF GASOLINE

Such characteristics of gasoline as ability to start an engine, ability to give proper acceleration, tendency towards crankcase dilution or vapor lock are determined entirely by the *physical* properties of gasoline. The tendency to knock, however, bears no direct relation to any physical property. With the completion of those researches on knock which led to the discovery of tetraethyl lead, it became apparent that the knocking tendency of gasoline is determined entirely by its chemical composition. Because of the outstanding importance of knock, a great deal of research began to be focussed upon the chemistry of gasoline and, as a result, the past fifteen years have seen, in this country, tremendous advances in our knowledge of hydrocarbon chemistry—advances which have led into many fields other than those of gasoline. The chemical nature of gasoline, therefore, warrants some discussion in this paper.

In the early days of refining, when the manufacture of gasoline was a simple distillation process, the actual chemical compounds present in any sample of gasoline were those that had been present as such in the crude oil from which the gasoline had been made. They varied greatly with the source of the crude oil, and they were outside the control of the refiner. They consisted, in general, in paraffins (normal and *iso*), naphthenes, and aromatics, with a preponderance of paraffins in gasoline made from, say, Pennsylvania crude oil, and with larger proportions of naphthenes occurring in gasoline made from California crude oil.

With the advent of "cracking," which in its original form was merely the breaking down by high temperatures and pressures of more complex hydrocarbons into those boiling in the gasoline range, another class of hydrocarbons, olefins or unsaturates, was added to the list, and added certain problems by virtue of their relative instability, and consequent tendency to form gum in storage. As recognition grew of the importance of the chemical nature of gasoline, and as technical knowledge developed of the thermodynamics and kinetics of hydrocarbon reactions, it has been found possible to alter profoundly the chemical nature of gasoline to produce greater and greater amounts of hydrocarbons of the types more desirable for different ultimate purposes. Figure 8 shows typical hydrocarbons of the different classes.

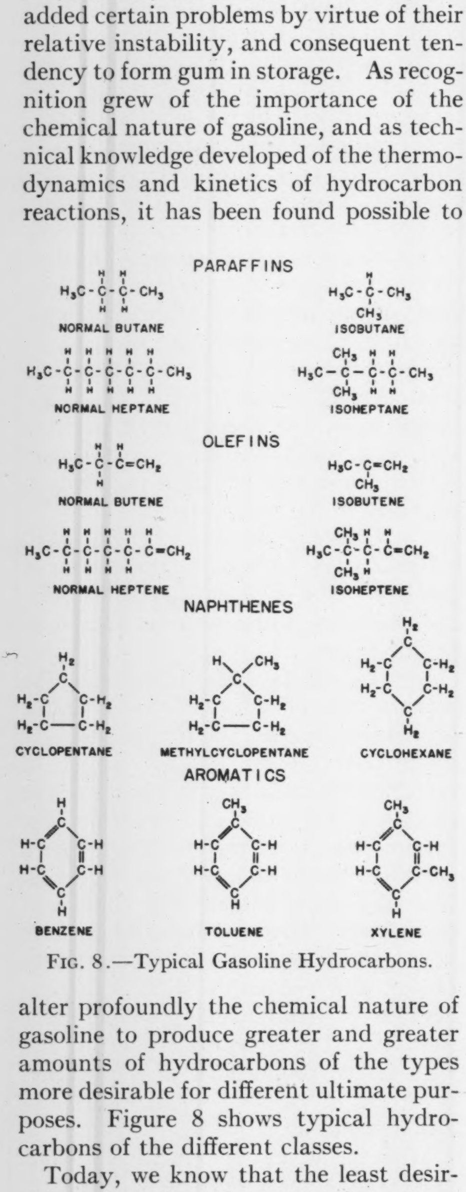
PARAFFINS
NORMAL BUTANE
ISOBUTANE
NORMAL HEPTANE
ISOHEPTANE
OLEFINS
NORMAL BUTENE
ISOBUTENE
NORMAL HEPTENE
ISOHEPTENE
NAPHTHENES
CYCLOPENTANE
METHYLCYCLOPENTANE
CYCLOHEXANE
AROMATICS
BENZENE
TOLUENE
XYLENE

FIG. 8.—Typical Gasoline Hydrocarbons.

Today, we know that the least desirable hydrocarbons for any engine use are the normal paraffins because of their extreme tendency to knock. We know that the *iso*paraffins are much superior to the normal paraffins and, in many cases, are excellent fuels. We know that olefins, particularly *iso*olefins, are excellent fuels for automotive use, especially under low-speed conditions. We know that naphthenes are moderately good automotive fuels, and that aromatics are excellent. We know that the distribution of the various types of hydrocarbon over the distillation range of motor gasoline affects profoundly the service performance of the gasoline. We know that for supercharged aircraft engines, many *iso*paraffins are excellent fuels for lean-mixture operation, and aromatics are effective under take-off conditions. And, as this knowledge has been attained, it has gradually become technically possible to alter the proportions of any one

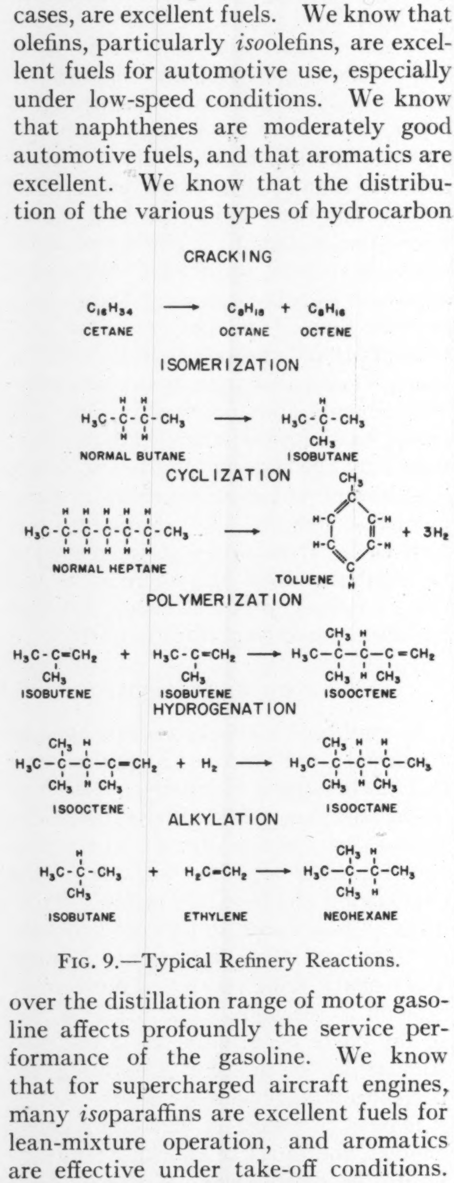
CRACKING
CETANE
OCTANE
OCTENE
ISOMERIZATION
NORMAL BUTANE
ISOBUTANE
CYCLIZATION
NORMAL HEPTANE
TOLUENE
+ 3H₂
POLYMERIZATION
ISOBUTENE
ISOBUTENE
ISOOCTENE
HYDROGENATION
ISOOCTENE
ISOOCTANE
ALKYLATION
ISOBUTANE
ETHYLENE
NEOHEXANE

FIG. 9.—Typical Refinery Reactions.

of these classes of hydrocarbons in gasoline. For example, the simple cracking reaction (Fig. 9) produces gasoline having a higher olefin content than straight-run gasoline. But the actual cracking reaction is never so simple. Under varying conditions of pressure and temperature, and particularly in the presence of catalysts, many other changes occur. The larger molecules of the heavy oils are broken into fragments of many sizes, including gases and carbon itself, and the fragments may undergo rearrangement in such ways that the types of hydrocarbon present in the finished gasoline may vary widely from the types present in the original crude oil. Even then, there is much that the refiner may do. The light gases may be built up into liquid hydrocarbons by such processes as polymerization and alkylation, the reactions being directed in such ways as to give maximum yields of specific hydrocarbon types. The crude gasoline (or naphtha) may again be subjected to temperature, pressure and catalysts (reforming), with the result that further rearrangement of the hydrocarbon molecules may take place. Treatment with catalysts may cause conversion of normal paraffins to the more desirable *iso*-paraffins (isomerization). It is even possible to fractionate out some of the normal paraffins, and by appropriate catalytic treatment convert them into aromatics. Accordingly, the petroleum refiner of today is approaching more and more the position of a synthetic organic chemist, and the gasoline of today is approaching more and more to the status of a blend of specific hydrocarbons, deliberately synthesized for various specific purposes.

AVIATION GASOLINE

So far, this paper has referred to aviation gasoline only incidentally. There have been several reasons for this casual treatment. In the first place, the volume of aviation gasoline has, until recently, been relatively so small in comparison to that of motor gasoline that it had during this period little effect upon the progress of the petroleum industry as a whole. Aviation demanded a stable straight-run gasoline, of lower vapor pressure through higher over-all volatility than motor gasoline, and an octane number which only in the past few years has exceeded 87. Such gasoline could be supplied by the refiner by fractionation of the straight-run gasoline made from selected crude oils, with the further addition of tetraethyl lead. More recently, the great increases in power obtainable from the supercharged aircraft engine by the use of high octane fuel began to bring the demand for octane numbers higher than could be obtained by the above simple process, and *iso*paraffins such as alkylate and *iso*octane began to be manufactured and added to aviation gasoline.

The war has increased tremendously the demand for aviation gasoline, and the demand for performance in military aircraft has brought current standards of antiknock value to the figure of 100 octane number, a figure which may well be exceeded as time goes on. The steps that have been taken to effect this result involve refining processes among those already discussed, but their exact nature and the extent of their development comes under the general shroud of military secrecy, and details must wait until after the war for full discussion.

IMPACT OF THE WAR ON GASOLINE

The war has affected profoundly the operations of practically every industry, and the petroleum refiner has not escaped its impact. In fact, in this war, gasoline has largely replaced smokeless powder as a propellant for high explosives, and has largely replaced both the army mule and the human foot as a means of transporting supplies and manpower. No single raw material appears to be as vital to the prosecution of a modern war as gasoline.

It has been necessary to expand enormously the production of high octane aviation gasoline, and this has meant not only the building of new plants designed to produce the *iso*paraffins and aromatics so necessary for 100 octane aviation gasoline, but the stripping of much motor gasoline of those constituents useful in aviation gasoline. At the same time, the necessity for curtailing motor gasoline consumption, partially because of transportation difficulties and partially to conserve rubber tires, while at the same time producing as much fuel oil as possible, has made it necessary for many refiners to alter their operations profoundly, so as to produce sometimes as little gasoline as possible instead of as much as possible. The desire of the Government to conserve raw materials has curtailed the amount of tetraethyl lead available for civilian use.

As a result, the trends of improving quality in civilian gasoline which have continued for so many years have been suddenly reversed. The average octane number of "house brand" gasoline has already dropped in six months from about 75 to 72, and may drop further. Over-all volatility may be affected. We are probably in "for the duration" for a completely abnormal operation of the petroleum industry, and for lowered quality as well as quantity in gasoline for civilian use. Military equipment will, of course, have high-quality gasoline provided, and the indications are that gasoline of adequate quality will be available for trucks, buses and other commercial vehicles, but the civilian will have to get along as best he can. Fortunately, the necessity for conserving tires will change civilian driving habits in such a way as to make maximum performance considerably less important than in the past, and the lowered quality will probably cause much less complaint than the lowered quantity.

POST-WAR GASOLINE

Any predictions concerning post-war gasoline, or any other phase of the post-war world, must be made with very considerable reservation. However, what post war gasoline *could be* is by this time fairly well established.

In the first place, it can be said with assurance that aviation gasoline will be much more important after the war than before, and that 100 octane number aviation gasoline, or better, will be the quality generally employed. No one can predict with certainty the part which aviation will play in post-war transportation, but it will undoubtedly be a far greater part than in the pre-war era, and will probably expand into the field of fast freight movement as well as the mail and passenger field. High power output and fuel economy will be of the utmost importance, and gasoline quality will, therefore, be as important as it is for military purposes.

But what of the future of motor gasoline? The tremendous demand for high octane aviation gasoline together with the program for synthetic rubber has brought about the erection of greatly increased catalytic cracking capacity, and this capacity undoubtedly can and will be used after the war to produce motor gasoline of high antiknock quality. Plants for producing the alkylate so necessary for aviation gasoline will have capacities probably much greater than the post-war demand for aviation gasoline, as great as they may be expected to be. Plants for producing toluene and other aromatics will probably be available. Furthermore, the research and development laboratories of the petroleum industry, always alert, have been profoundly stimulated by the war effort and can be counted on for processes which are improvements over what we have today. I believe it to be conservative to say that the petroleum industry will be in a position to produce motor gasoline of much higher effective antiknock value than that which was produced in 1941. One might guess that gasoline approximately equivalent to the premium grade of 1941 (say, 80 octane number) could be produced as a house brand gasoline, and that premium gasoline having an effective antiknock value of between 90 and 100 octane number could be produced.

Will such gasoline be produced and marketed? The answer to this question depends largely upon the automotive engineer. The automotive industry finds itself at present in a position unique in its history. Heretofore, under the continued pressure of getting out yearly models, desirous of reusing as many tools, dies, etc., from previous models as might be feasible, and doubtful of public acceptance to too radical a change in car design, the automotive industry has made all changes conservatively and gradually. In fact, the saying is that the only reason that engines are still in the front of the car has been that no one could figure out a way to move them to the rear gradually! Now, the situation is that there are no yearly models, or production of cars of any kind, which gives the design engineer a breathing space in which to plan far-reaching improvements. Tools and dies will have been largely scrapped long before automobile manufacture can begin again. Metals, such as aluminum and magnesium, will be available in enormous quantity and at reduced price. New and better plastics will be available. Coupled with this will be a car-hungry public. What will the answer be? As far as body design goes, one can look forward with confidence to radical changes, but what of the engine? Will we have an engine of extremely high compression ratio, and consequent high thermal efficiency? Will there be a small supercharged engine capable of producing the necessary power with savings in weight and size? Will there be an engine of very moderate speed range equipped with automatic transmission? Will there be an engine embodying principles quite different from the conventional engine of today? No one can answer these questions, but it appears certain that no real developments along these lines can be expected unless the petroleum industry does its share by improving gasoline quality.

Are we reasoning in a circle in saying that improvement in the engine of the future depends upon the gasoline of the future, and that the gasoline of the future depends upon the engine of the future? No more so than has been true in the past; the two industries have always advanced together and must continue to do so. The only difference is that heretofore progress has been made by a steady series of small advances, made first by one industry and then by the other, or by both together; now, the war has brought about the possibility of fairly abrupt and quite substantial advances in technical progress. There are many serious problems, economic and technical, in such progress, but I cannot believe them to be insuperable, and I shall be greatly surprised if the best talent of both industries is not now giving its consideration to the problems of post-war transportation. And may they always work in closest contact, for they are inseparable parts of the same great industry, the industry of transportation, and must progress simultaneously, if the public is to receive the more efficient, more economical, more comfortable, and safer transportation which appears to be definitely technically and economically possible.

THE PHYSICAL PROPERTIES OF STEEL WIRE AS AFFECTED BY VARIATIONS IN THE DRAWING OPERATIONS

By Howard J. Godfrey[1]

Synopsis

This investigation was carried out on 0.67 per cent carbon patented wire drawn 40, 30, 20, and 10 per cent reduction per draft, and a 0.27 per cent carbon patented rod drawn 40, 30, and 20 per cent reduction per draft. The physical tests, which included tension, torsion, bending and bending fatigue, were made at a number of stages of wire drawing from the patented condition to 97 per cent reduction. The physical properties of hand-straightened and machine-straightened wire were compared.

The tensile strength and bending properties were found to be changed considerably by the method of wire drawing. The lower rates of drawing resulted in improved bending properties and lower tensile strength. The physical properties of the 0.67 per cent carbon material were affected to a greater extent by the rate of drawing than the 0.27 per cent carbon wire. In general the elongation and reduction of area as determined in the tension test were improved by a low rate of drawing. The fatigue properties were affected by the rate of drawing in the same manner as the tensile strength. The number of bends and type of fracture obtained in the bend test are believed to be a measure of the toughness of steel wire. The amount of wire drawing that resulted in the best combination of strength, ductility, toughness, and fatigue properties was between 75 and 80 per cent reduction.

The influence of variations in the drawing operations on some of the physical properties of steel wire has long been appreciated by the wire industry. The purpose of this investigation was to study in detail the physical properties of wire drawn according to a number of drafting schedules and to correlate the physical properties as determined by various types of test methods. The method of wire drawing resulting in the best combination of physical properties could then be established.

The tension and torsion tests are usually employed to govern the quality of finished wire. In order to provide additional information as to the quality of wire, another method of testing was developed. First suggested by Bonzel (**1**)[2] in his book "Steel Wire," a bend test was described in a previous paper (**2**) which when properly made seemed to indicate the toughness of steel wire. The further development of this bend test has been included in this investigation.

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[1] Development Engineer, John A. Roebling's Sons Co., Trenton, N. J.

[2] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 526.

TABLE I.—CHEMICAL COMPOSITION OF STEEL.

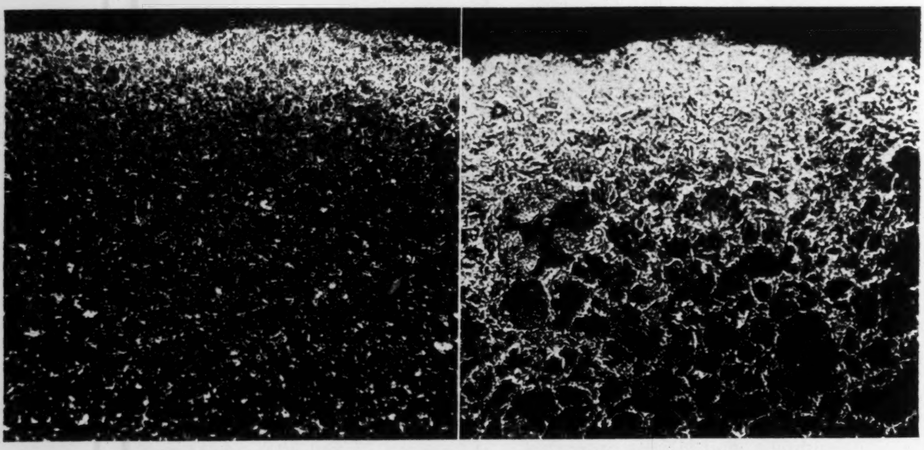
Carbon, per cent	Manganese, per cent	Phosphorus, per cent	Sulfur, per cent	Silicon, per cent
0.67	0.84	0.021	0.027	0.19
0.27	0.50	0.014	0.031	Trace

Among the more recent investigations on steel wire, are those of Gill and Goodacre (5, 6, 7, 8), who have studied the physical properties of both decarburized wire and wire free from decarburization. In the above investigation, the diameter of the wire tested was always the same, and the amount of wire drawing was varied by changing the size of the original patented material. In this present investigation, the wire of a given carbon content was drawn from one original coil and tested at various stages of wire drawing.

MATERIALS

Two materials were used, a 0.67 per cent and a 0.27 per cent carbon steel, the chemical compositions of which are presented in Table I. This analysis was made on samples of patented material from which the wire was drawn.

All of the 0.67 per cent carbon wire came from one coil of $\frac{1}{4}$-in. diameter hot-rolled rod which was drawn to 0.218 in. in diameter and double lead patented at 1550 to 900 F. This patented wire was then drawn 40, 30, 20, and 10 per cent reduction per draft. For the 40 per cent group, only two sizes were supplied since breakage occurred during wire drawing on the third draft. The 0.27 per cent carbon wire was drawn 40, 30, and 20 per cent reduction per draft from a 0.220-in. diameter patented rod. A number of sizes of wire representing each rate of drawing were furnished by the wire mill in both coils and machine straightened.



0.67 per cent Carbon 0.27 per cent Carbon

FIG. 1.—Patended Structure of 0.67 per cent and 0.27 per cent Carbon Steel (× 100).

The microstructure of the 0.67 per cent carbon and 0.27 per cent carbon steel after the patenting heat treatment are shown in Fig. 1. At the surface of both of these patented materials there is a distinct decarburized layer. Since all the wire of a given carbon content was drawn from the same coil, the effect of the decarburized surface would be the same throughout. Any differences in the physical properties could therefore be attributed to the method of wire drawing.

METHODS OF TESTING

The tension tests were carried out on a 60,000-lb. capacity Baldwin-Southwark hydraulic testing machine and a 2000-lb. capacity Amsler hydraulic testing machine. The tensile strength, elongation in 10 in., and the reduction of area were determined. On some of the tension specimens the ductility indices as described by MacGregor (3) were also determined. The ductility indices separate the uniform and necking phases which occur during the tension test. The uniform strain occurs between zero load and maximum load, and the necking strain occurs between the maximum load and the breaking load. The uniform ductility index I_u and the necking ductility index I_n are determined from the following equations:

$$\text{Uniform ductility index } I_u = \log \frac{A_o}{A_u}$$

$$\text{Necking ductility index } I_n = \log \frac{A_u}{A_b}$$

where:

A_o = minimum area at zero load,

A_u = minimum area at maximum load, and

A_b = minimum area after fracture.

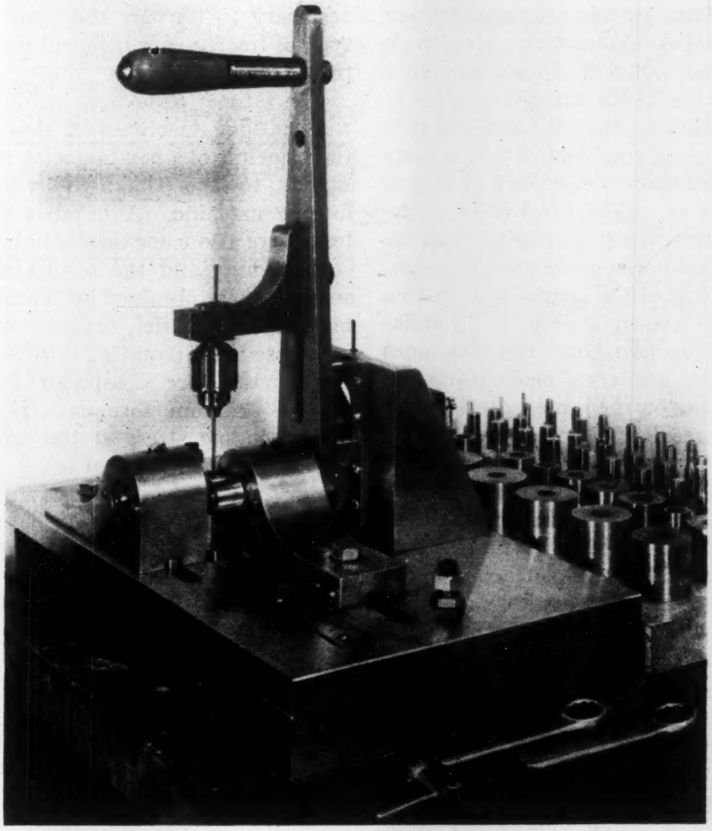


FIG. 2.—Bend Testing Machine for Wire.

The above values may be obtained by measuring the least diameter of the test specimens at the three stages of the test.

The torsion test, as a routine test, often as an acceptance test, is peculiar to the wire industry. The torsion tests were made using a gage length equal to 100 wire diameters and a speed of testing of 60 twists per minute with a tension on the specimen equal to approximately 1 per cent of the tensile strength.

The bending tests on the 0.67 per cent carbon wire were carried out on a wire-bending machine as described in a previous paper (2). The bend tests on the 0.27 per cent carbon wire were made on another machine of more recent design. A photograph of the latter bend testing machine is shown in Fig. 2. Fundamentally, the two bend test machines are identical and tests made using the two machines on the same material gave the same results. The test, on either machine, consists of bending the wire specimen back and forth over rolls by moving the bending arm through an angle of 180 deg. until fracture occurs. The wire is held in a grip attached to the bending arm and weights are suspended from the lower end of the wire. The amount of tensile stress on the wire specimen due to the suspended weights is 1 per cent of the tensile strength of the wire. The center of rotation of the bending arm is adjustable for any diameter roll and is set at a point 0.7 R above the centerline of the rolls, where R = radius of the rolls. In this position the vertical movement of the wire during the test is held to a minimum. In order to correlate the bending properties of wires of different diameters, the ratio between the roll diameter and the wire diameter should be the same. A roll diameter - wire diameter ratio of 10 was used in these experiments. If a lower ratio than 10 is used, the number of bends decreases and it becomes difficult to differentiate between the bending properties. A larger ratio would only serve to prolong the length of the test. Whenever rolls of exactly 10 wire diameters were not available, tests were made on two sets of rolls of the approximate size and the values interpolated for the ratio of 10. The number of 180-deg. reverse bends necessary to fracture the wire and the type of fracture are believed to indicate the toughness of wire.

The fatigue tests were made on two wire fatigue machines (2) designed on the principle of a rotating wire strut and similar to the Haigh-Robertson wire fatigue machine. A number of check tests were also made on a Haigh-Robertson machine and the results were the same as those obtained on the two other machines. In brief, the wire specimen is stressed as a rotating wire strut and the stresses are computed from the "Euler" column formula. The maximum bending stress at the mid-length of the specimen is equal to:

$$f = \frac{\pi^2}{360} \theta \frac{d}{L} E$$

where:

f = bending stress in pounds per square inch,

θ = inclination at end of specimen in degrees,

d = diameter of wire in inches,

L = length of wire in inches, and

E = modulus of elasticity in pounds per square inch.

Since the maximum stress occurs at the center of the specimen, all fractures occur at this point, thereby providing a method of fatigue testing in which the gripping devices have no effect on the test results. The specimen for a fatigue test must be straightened before testing; otherwise vibrations will be set up during the test. The tensile properties of the wire used for the fatigue tests were changed by the machine-straightening operation. However, a number of tests were made comparing the fatigue properties of machine- and hand-straightened wire and in all cases the results were within the normal variation of this material. The modulus of elasticity was assumed to be 30,000,000 psi. for all the material tested.

TEST RESULTS

Effect of Straightening Process:

In general the tensile strength of the machine-straightened wire is less than that of the hand-straightened material and the loss in strength due to the machine-straightening process diminishes as the size of the wire decreases. Examples of the effect of the straightening process are shown in Fig. 3, where autographic load-elongation curves of a 0.67 per cent carbon wire are presented. The tensile strength has been reduced by the machine-straightening operation but at the same time the elongation has been increased considerably. The effect of the straightening process on the tensile strength of the 0.67 per cent and 0.27 per cent carbon wire drawn 40 per cent per draft is shown in Table II.

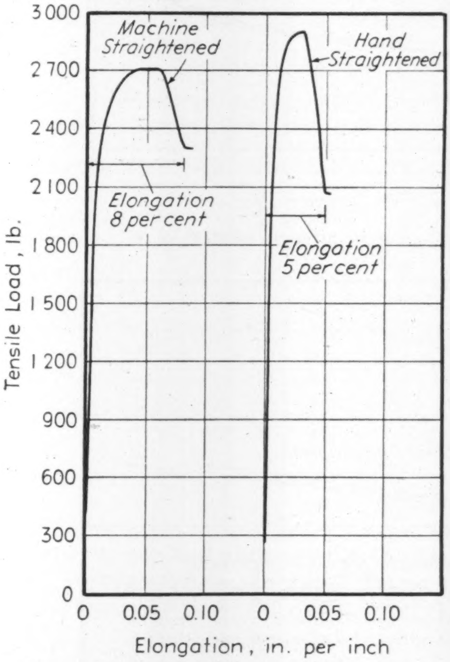


FIG. 3.—Effect of the Straightening Process on the Tensile Properties of 0.67 per cent Carbon Steel Wire, diameter = 0.129 in.

The fatigue properties of both hand- and machine-straightened material are also presented in Table II. These tests indicated that the endurance limit could be determined on the machine-straightened wire with the necessary accuracy even though the tensile strength of this material was somewhat lower than the hand-straightened material. All other fatigue properties reported in this paper were therefore determined on machine-straightened wire.

TABLE II.—TENSILE STRENGTH AND ENDURANCE LIMIT OF HAND- AND MACHINE-STRAIGHTENED WIRE.

Material	Diameter, in.	Draft	Tensile Strength, psi.		Endurance Limit, psi.	
			Hand	Machine	Hand	Machine
0.67 per cent carbon	0.218	Rod	161 000	158 000	35 500	37 000
	0.169	1	201 000	185 000	52 000	49 000
	0.1315	2	230 000	216 000	59 000	60 000
0.27 per cent carbon	0.1025	3	143 000	133 000	52 000	54 000
	0.079	4	164 000	153 000	56 000	58 000
	0.0615	5	182 000	175 000	62 000	62 000
	0.0468	6	211 000	206 000	66 000	68 000
	0.0368	7	244 000	237 000	72 000	74 000

Tensile Strength:

The effect of the rate of drawing on the tensile strength of the 0.67 per cent and 0.27 per cent carbon wire is shown in Fig. 4. The amount of cold working is expressed by the percentage reduction of area by wire drawing and is determined from the following equation:

Percentage Reduction of Area

$$= \frac{A_o - A_f}{A_o} \times 100$$

where:

A_o = initial area of patented material, and

A_f = area of cold-drawn wire at any stage of wire drawing.

Using the above expression as a measure of the amount of wire drawing we find that the rate of increase in tensile strength is greatest in the latter stages of wire drawing.

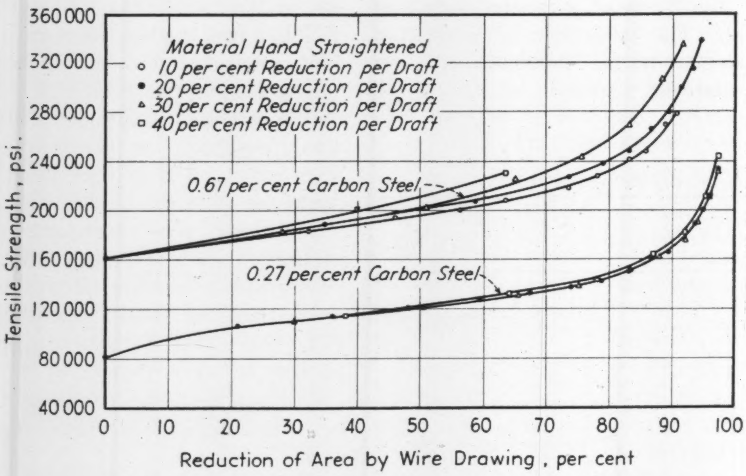


FIG. 4.—Effect of Wire Drawing on the Tensile Strength of Steel Wire.

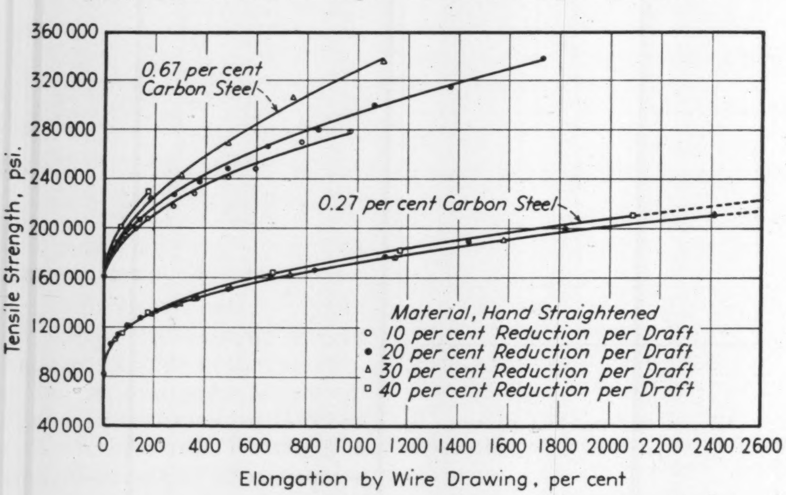


FIG. 5.—Effect of Wire Drawing on the Tensile Strength of Steel Wire.

The effect of the rate of drawing on the 0.67 per cent carbon wire is very definite. A higher rate of drawing produces a higher tensile strength for any given amount of wire drawing. The effect of the rate of drawing on the 0.27 per cent carbon wire was rather small, the material drawn 40 per cent reduction per draft being only slightly stronger than the material drawn 20 and 30 per cent reduction per draft.

Bonzel (1) has suggested that the amount of cold working by wire drawing could be expressed by the percentage elongation. This value is computed by the following equation:

Percentage Elongation

$$= \frac{A_o - A_f}{A_f} \times 100$$

The above equation expresses the percentage increase in the length of the original wire. The tensile strength is related to the percentage elongation by wire drawing in Fig. 5. In this relationship, the rate of increase in strength is greatest during the initial stages of wire drawing, in direct contrast to Fig. 4, where the tensile strength is related to the reduction of area. The effect of the rate of drawing, however, is not changed by this method of expressing the amount of cold working.

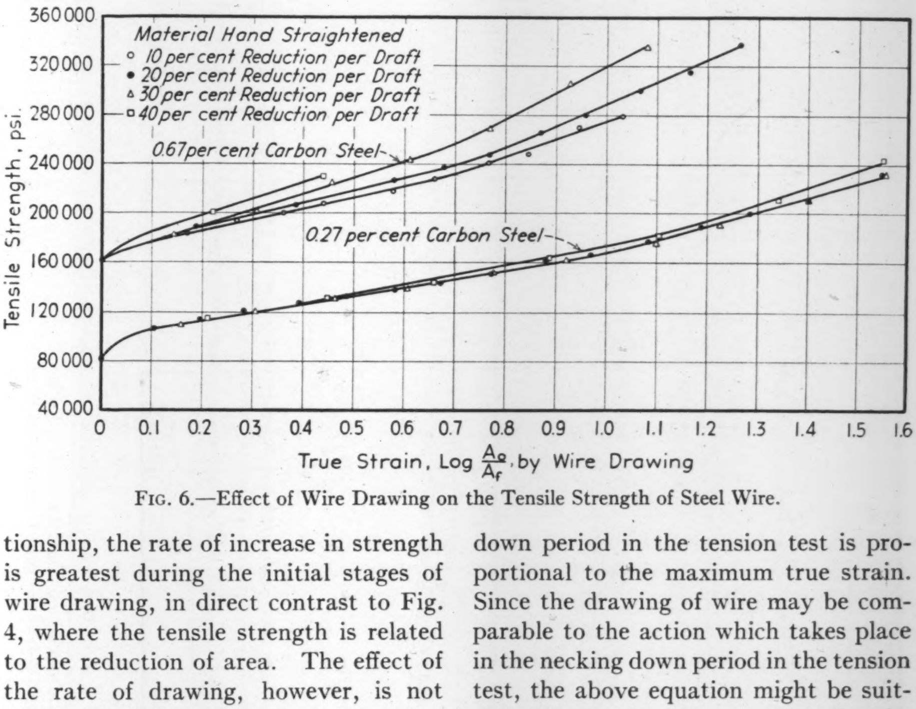


FIG. 6.—Effect of Wire Drawing on the Tensile Strength of Steel Wire.

A third method of expressing the amount of cold working by drawing is presented in Fig. 6, where the tensile strength is related to the true strain. The equation for the amount of maximum true strain or true reduction of area in a simple tension test for a uniform cross-section is expressed by MacGregor (3) as follows:

$$\text{Maximum True Strain } \epsilon = \log \frac{A_o}{A_f}$$

In the tension test it has been found that the true stress-strain curve becomes a straight line from the value of the average true stress corresponding to the maximum load up to the breaking load. This would mean that the amount of work hardening done during the necking down period in the tension test is proportional to the maximum true strain. Since the drawing of wire may be comparable to the action which takes place in the necking down period in the tension test, the above equation might be suitable criterion for the expression for the amount of cold work done by wire drawing. In Fig. 6 the tensile strength increases in direct proportion to the amount of true strain up to a certain amount of drawing, after which the tensile strength increases at a somewhat greater rate in the latter stages of wire drawing.

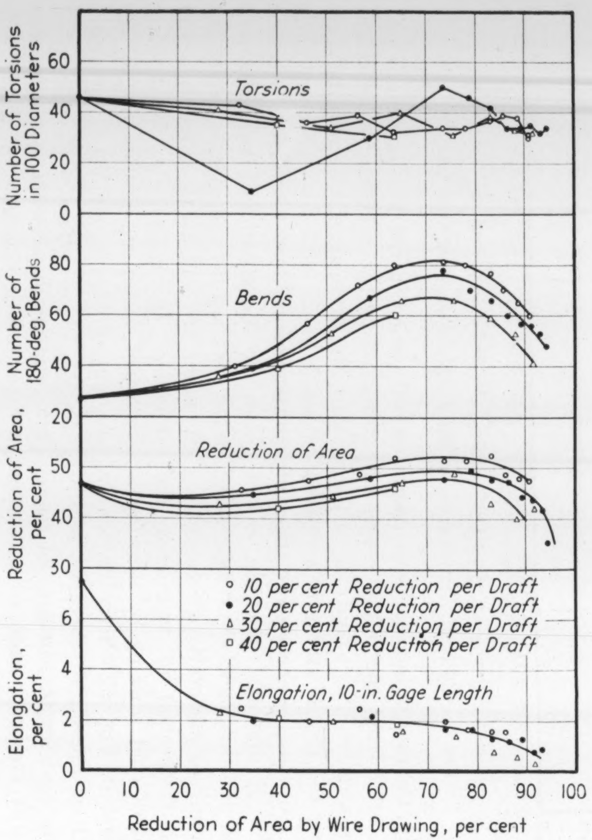


FIG. 7.—Effect of Wire Drawing on Torsions, Bends, Reduction of Area, and Elongation of 0.67 per cent Carbon Steel Wire, Hand Straightened.

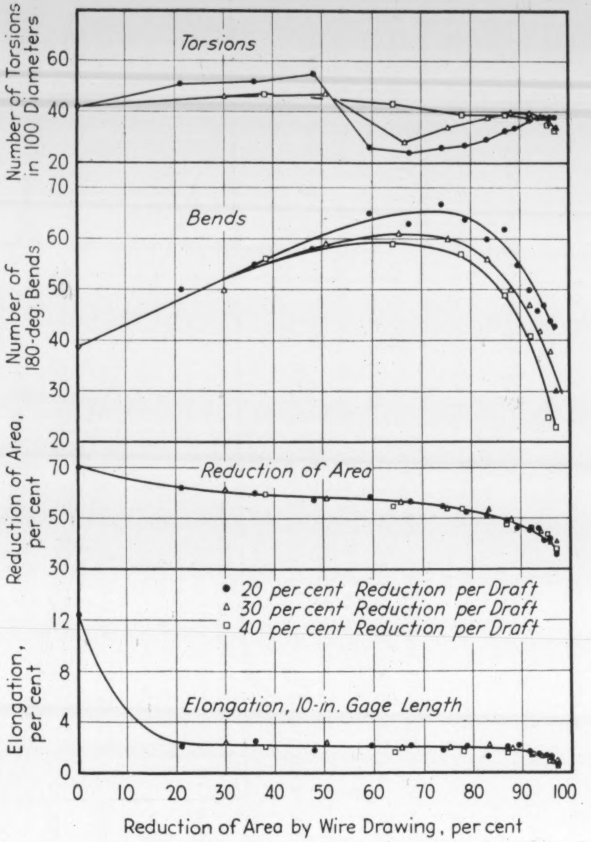


FIG. 8.—Effect of Wire Drawing on Torsions, Bend, Reduction of Area, and Elongation of 0.27 per cent Carbon Steel Wire, Hand Straightened.

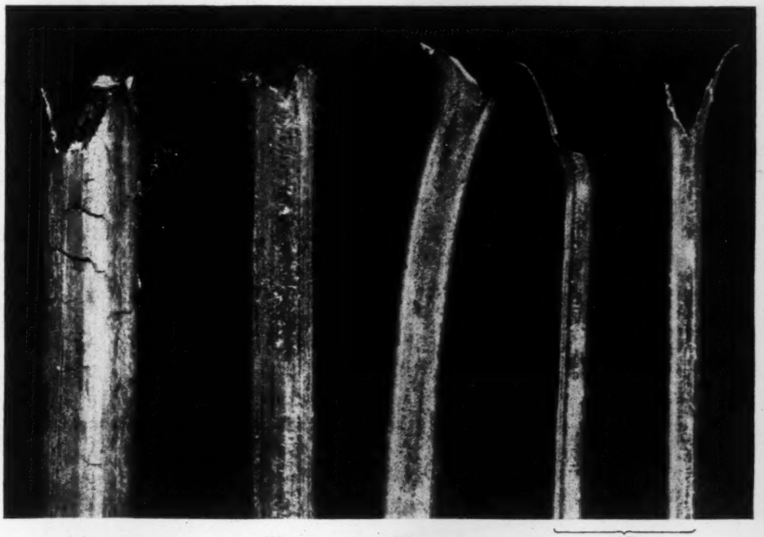
Elongation, Reduction of Area, Torsion and Bending:

The effect of the rate of drawing on the elongation in 10 in., reduction of area, torsion, and bending properties of the 0.67 per cent and 0.27 per cent carbon wire is presented in Figs. 7 and 8, respectively. For the 0.67 per cent carbon wire the lower rate of drawing resulted in higher elongation, particularly in the latter stages of wire drawing. The difference, however, was not very marked. The effect of the rate of drawing on the elongation of the 0.27 per cent carbon wire was negligible.

The reduction of area of the 0.67 per cent carbon wire was affected to a large degree by the rate of drawing but no differences could be found on the 0.27 per cent carbon wire. The lower rates of drawing on the high-carbon wire resulted in a higher reduction of area. The total amount of wire drawing had a decidedly different effect on these two materials. The reduction of area of the 0.67 per cent carbon wire first decreased and then increased with wire drawing up to about 75 per cent reduction and then decreased rapidly. The reduction of area of lower carbon material, however, gradually decreased with wire drawing up to about 80 per cent reduction, after which the ductility decreased rapidly.

The results of the torsion tests, Figs. 7 and 8, show that except for one specimen, all of the 0.67 per cent carbon material had at least 30 torsions in 100 wire diameters. The rate of drawing and the total amount of wire drawing did not have any consistent effect on the torsional properties.

The bending properties of the 0.67 per cent and 0.27 per cent carbon steel wire are also presented in Figs. 7 and 8. A decrease in the rate of drawing on the 0.67 per cent carbon wire resulted in a considerable improvement in the bend-



(*a*) 58.6 per cent (*b*) 78.9 per cent (*c*) 86.5 per cent (*d*) 94.5 per cent

Percentage Reduction by Wire Drawing.

FIG. 9.—Bend Test Fractures of 0.67 per cent Carbon Wire Drawn 20 per cent Reduction per Draft.

ing properties. The drawing schedule did not have such a marked effect on the 0.27 per cent carbon wire except in the latter stages of wire drawing. The bending properties improved with wire drawing up to about 72 per cent reduction and then decreased very rapidly. The characteristics of the fracture obtained in the bend test also indicated the toughness of the material. Figure 9 shows a number of bend test fractures obtained on the 0.67 per cent carbon wire drawn 20 per cent per draft. Specimens *a* and *b*, which were cold drawn to a total of 58.6 per cent and 78.9 per cent reduction, respectively, have rather stubby fractures. Specimen *c*, drawn 86.5 per cent reduction, has a long slivery fracture with a tendency to form longitudinal cracks. Specimens *d*, drawn 94.5 per cent reduction, have a decided tendency to fracture in the longitudinal direction. The 0.27 per cent carbon wire had only a slight tendency to split in the longitudinal direction even though it was cold drawn more than the high-carbon wire.

In order to investigate further any other possible means by which to determine more accurately a true measure of the physical characteristics of wire, a number of measurements were made during the tension test to determine the true measure of ductility as expressed by MacGregor (3). An example of the results of these measurements are presented in Fig. 10 where the true uniform strain and true necking strain are compared to the usual reduction of area values for the 0.27 per cent carbon wire drawn 20 per cent per draft. The uniform strain for cold-drawn steel wire is found to be a small percentage of the true maximum necking strain. It should be noted that the uniform strain is practically a constant for the cold-drawn wire whereas the necking strain decreases gradually with wire drawing and the curve is very similar to the reduction of area curve. This true necking strain for the 0.67 per cent carbon wire was also proportional to the reduction of area as determined by the tension test.

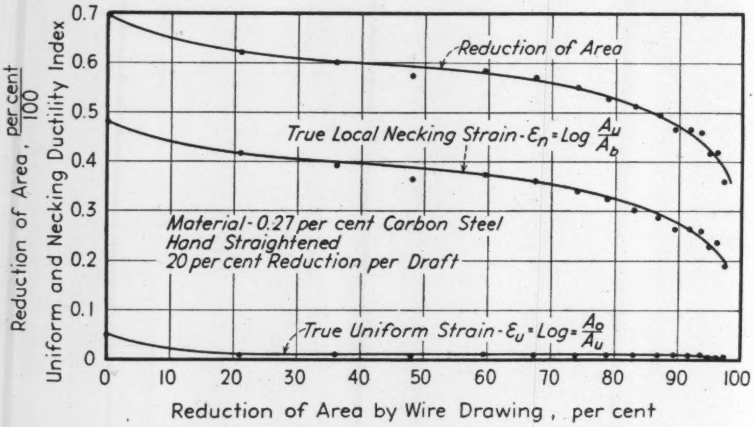


FIG. 10.—Effect of Wire Drawing on the Ductility Indices.

Fatigue Tests:

The relation between the fatigue and tensile properties is presented in Fig. 11. These results show that for the same tensile strength the lower carbon material has a considerably higher endurance limit. For an endurance limit of 60,000 psi., the tensile strength of the 0.27 per cent carbon wire is only 160,000 psi., as compared to 230,000 psi. for the 0.67 per cent carbon wire even though both materials were definitely decarburized.

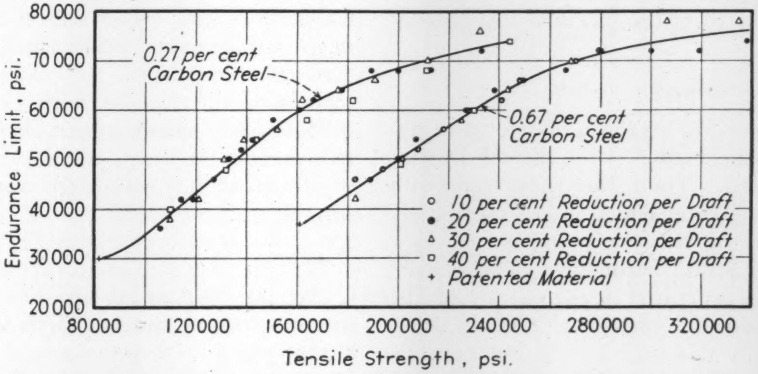


FIG. 11.—Relation Between Endurance Limit and Tensile Strength of Steel Wire.

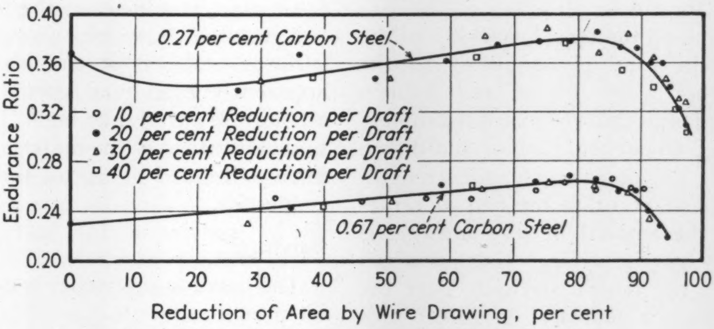


FIG. 12.—Effect of Wire Drawing on the Endurance Ratio.

The effect of wire drawing on the endurance ratio is presented in Fig. 12. As previously shown, the endurance limit of the hand-straightened wire was practically the same as that of the machine-straightened wire. The endurance ratio was therefore calculated by dividing the endurance limit of the machine-straightened wire by the tensile strength of the hand-straightened wire. The endurance ratio increases very gradually up to approximately 80 per cent reduction by wire drawing and then decreases rapidly. Since the endurance ratio is approximately the same for each drawing schedule, the endurance limit is affected by the rate of drafting in the same manner as the tensile strength.

DISCUSSION OF TEST RESULTS

Effect of Straightening Process:

The method used in straightening wire was found to have a marked effect on the tensile properties. The tensile strength of the machine-straightened wire was less than that of the hand-straightened material but the ductility as measured by the elongation was improved. The above effects were more pronounced on the larger diameter wires. This indicates that the loss in strength and increase in ductility might be due to the reverse bending which takes place in the rotary machine straightener. Since the endurance limit is only a relatively small percentage of the tensile strength, it was not affected by the loss in strength of the machine-straightened wire.

Tensile Strength:

The tensile strength of steel wire was in general higher as the rate of drawing increased. The higher rates of drawing have a greater effect on the total cross-section of the wire, and result in a greater strain hardening. The amount of heat generated in the die would also be greater for the higher rates of drawing and this increase in temperature would tend to raise the tensile strength of the wire. Temperature would also account for the small effect of the rate of drawing on the tensile strength of the low-carbon wire as compared to the high-carbon wire. Püngel and Hünlich (4) have found that up to approximately 250 C. a 0.66 per cent carbon cold-drawn steel wire will increase in tensile strength.

The most accurate method of representing the amount of wire drawing is probably by true strain. Using this as a measure, the tensile strength after the first reduction increases in direct proportion to the amount of wire drawing. If this straight-line relationship holds true for all cold-drawn wire it would be quite advantageous to use the relationship to determine the amount and method of drawing to obtain the necessary tensile strength of a finished wire. The change in the rate of increase in tensile strength in the final stages of wire drawing might be explained by the increase in the amount of preferred orientation (2). Since the crystal orientation is such that the face diagonal of the cube, that is the [110], lies parallel to the wire axis, a change in the rate of increase in strength would be expected.

Ductility:

The rate of drawing had considerably more effect on the elongation and reduction of area of the high-carbon than the low-carbon wire. The reason for this might also be explained by the temperature effect during wire drawing. The increase in the ductility with a decrease in the rate of drawing may also be due to the more uniform deformation which would take place within the wire-drawing die.

Torsion:

The rate of drawing and the amount of wire drawing did not have any consistent effect on the torsion test results. The torsion test is probably affected by the general fibering of the grains and the surface condition of the wire. The test results, however, show that the surface condition of the material was apparently good even after an extreme amount of wire drawing. The decarburized surface of the material may also have a decided effect on the torsional properties.

Bending:

The bending properties were found to be more sensitive to the rate of drawing than any other measure of the physical characteristics of steel wire. Even though the ductility of the 0.27 per cent carbon wire was not changed materially by the rate of drawing, the bending properties were definitely affected. The improvement in the bending properties by a low rate of drawing may be attributed to a more uniform fibering of the grains as a whole. The fact that the number of bends decrease after approximately 72 per cent reduction by wire drawing indicates that the beneficial effect of the initial fibering is being offset by the loss in ductility and by the directional properties which are characteristic of materials which have excessive preferred orientation. The type of fracture obtained in the bend test on wire also indicates that longitudinal planes of weakness are formed in wire as the amount of wire drawing exceeds certain limits.

Fatigue:

The fatigue properties of the 0.67 per cent and 0.27 per cent carbon wire were considerably lower than would be found on a wire free from decarburization. From previous tests (2), the endurance limit of a 0.67 per cent carbon wire free from decarburization should have been approximately 32 per cent of the tensile strength instead of 25 per cent. The lower endurance limit of a decarburized material is due to the fact that the maximum bending stresses on the fatigue specimen occur at the surface of the wire where the strength of the wire due to decarburization is at a minimum. The decarburization of the 0.27 per cent carbon wire would have less effect on its fatigue properties since the difference in strength of the decarburized surface and the strength of the core of the wire would be less. The rapid drop in the endurance ratio after approximately 80 per cent reduction by wire drawing may be due to the variation in tensile strength over the cross-section of the wire. The greatest amount of preferred orientation has been found to occur at the center of the wire, which would result in a higher tensile strength at this section. Since the tensile strength is the average strength of the cross-section and the fatigue limit is dependent upon the strength of the surface of the wire, a lowering of the fatigue ratio would be expected.

CONCLUSIONS

1. The tensile strength of cold-drawn wire is reduced by the machine-straightening operation and its ductility as measured by the elongation is increased.

2. The tensile strength of cold-drawn wire is materially affected by the rate of drawing. The greater the reduction in area per draft, the higher the tensile strength will be for the same total amount of wire drawing.

3. The 0.67 per cent carbon wire was affected to a greater extent by the rate of drawing than the 0.27 per cent carbon wire.

4. The reduction of area as determined by the tension test increased as the rate of drawing decreased on the 0.67 per cent carbon wire. The 0.27 per cent carbon wire was not affected in this respect by the rate of drawing.

5. The elongation in 10 in. was slightly improved by a low rate of drawing.

6. The rate of drawing had no consistent effect on the torsional properties of the material investigated.

7. The bending properties were considerably improved by reducing the rate of drawing. The bend test indicates that there is an optimum amount of wire drawing which will result in a maximum toughness for cold-drawn wire. The number of bends to cause fracture and the appearance of the fracture obtained in the bend test are believed to be a measure of the toughness of steel wire.

8. The rate of drawing affected the endurance limit in the same manner as the tensile strength. The endurance limit of the decarburized material was considerably lower than has been found for wire free from decarburization.

9. The endurance ratio increased slightly up to approximately 80 per cent reduction by wire drawing. The endur-

ance ratio then decreased rapidly with further wire drawing.

10. The best combination of strength, ductility, toughness, and fatigue properties resulted from a total amount of wire drawing of between 75 and 80 per cent reduction.

Acknowledgments:

The main part of this investigation was carried out at the Fritz Engineering Laboratory at Lehigh University, and the author wishes to express his appreciation to the Laboratory staff, which includes Prof. Hale Sutherland, Director, and Dr. Bruce Johnston, Assistant Director, for their valued assistance.

The author also wishes to express his sincere thanks to C. W. Meyers of the American Steel and Wire Co. for his generous cooperation and for the materials used in this investigation; to L. H. Winkler of the Bethlehem Steel Co. for his continued advice and assistance in making part of the torsion tests.

The author wishes to thank the John A. Roebling's Sons Co. for the permission to complete this investigation in their Development Engineering Laboratory, which is under C. M. Jones, Director of Engineering, and Dartrey Lewis, Chief Development Engineer.

REFERENCES

(1) Maurice Bonzel, "Steel Wire," pp. 185–218, Engineers Book Shop, New York, N. Y. (1935).

(2) H. J. Godfrey, "The Fatigue and Bending Properties of Cold Drawn Steel Wire," *Transactions*, Am. Soc. Metals, Vol. XXIX, March, 1941, p. 133.

(3) C. W. MacGregor, "The Tension Test," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 508 (1940).

(4) W. Püngel and R. Hünlich, "Effect of Tempering on Elastic Properties of Steel Wire," *Mitteilungen Kohle und Eisenforschung*, Vol. 2, pp. 185–188 (1940).

(5) E. T. Gill and R. Goodacre, "Some Aspects of the Fatigue Properties of Patented Steel Wires," *Journal*, Iron and Steel Inst., No. 2, Vol. CXXX, pp. 293–323 (1934).

(6) E. T. Gill and R. Goodacre, "Some Aspects of the Fatigue Properties of Patented Steel Wires," *Journal*, Iron and Steel Inst., No. 2, Vol. CXXXII, pp. 143–177 (1935).

(7) E. T. Gill and R. Goodacre, "Some Aspects of the Fatigue Properties of Patented Steel Wires," *Carnegie Scholarship Memoirs*, Vol. XXV, pp. 93–110 (1936).

(8) R. Goodacre, "Some Aspects of the Fatigue Properties of Patented Steel Wires," *Carnegie Scholarship Memoirs*, Vol. XXV, pp. 111–139 (1936).

DISCUSSION

Mr. L. H. Winkler.[1]—There are one or two points brought out in this excellent paper which justify further discussion.

First, I want to congratulate Mr. Godfrey on his paper and especially on the mechanical ingenuity displayed in developing a bend test procedure and the equipment described. Bend tests on wire heretofore have not been by any means satisfactory; quite possibly the mechanical equipment that was available at the time for making such bend tests was at fault. I think Mr. Godfrey has made a notable contribution to the testing of wire. His perfection and improvement of bend tests appear to have accomplished a great deal toward providing results that are more consistent than has generally been obtained in past work.

Up until now many of us have regarded with skepticism bend test requirements and bend test results on wire. Judging from the amount of work which Mr. Godfrey has done to date and from the results reported in his paper, it would appear that we may begin to look upon bend tests with more assurance, particularly if they are carried out in a manner similar to that which he describes.

In practical experience we find that the rate of drawing does influence mechanical properties of wire. Those of us who have been associated with wire drawing have found that higher percentages of reduction per draft will usually result in higher tensile strength in the wire than would be the case if less reduction per draft (and, therefore, more drafts) were employed for the same given total reduction. At the same time, in actual practice we have also noted a corresponding reduction in the torsion values when the number of reductions have been reduced by virtue of greater reduction per draft. Upon reading Mr. Godfrey's paper I was surprised that this effect on the torsion values was not definitely borne out by results obtained in his research. It is possible that this may be accounted for by some detail in the preparatory heat treatment of the stock from which the wire was drawn; doubtless further work, which I expect he will pursue, will throw more light on this question.

Mr. Godfrey's work shows that apparently for a given size and type of wire a total reduction of between 70 and 75 per cent results in the most desirable combination of physical characteristics. I should like to call attention to the fact that this may apply quite specifically only to the size ranges and the kind of wire covered by Mr. Godfrey's experimental work. The optimum percentage of reduction, I believe, will change with the size of the finished wire and also will depend upon the kind of wire that is to be produced. That is, although the best reduction may be between 70 and 75 per cent for these relatively large diameters of wire, it may be advisable to increase the percentage of total reduction in the manufacture of smaller sizes. For example, in the case of a similar grade of wire about 0.015 in. in diameter, a total amount of reduction of about 85 per cent may produce better results. Furthermore, the total amount of reduction is quite definitely dependent upon the details of the preparatory heat treatment, the number of drafts, the size of the wire, and especially upon the kind of wire that is to be produced. The point that I wish to make is that there is no one optimum percentage of reduction which will cover all sizes and kinds of wire currently produced in the full range of steel wire mill products, beginning with low carbons and ending with very high carbons; this is probably obvious and while I realize that Mr. Godfrey may be in agreement with this statement I think it is important to emphasize that point.

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[1] Metallurgical Engineer, Bethlehem Steel Co., Inc. Bethlehem, Pa.

Mr. J. H. Foote.[2]—There are two points brought out in this paper which might merit further study.

In the application of steel strand to overhead tower lines as ground wires, the problem of fatigue due to conductor vibration arises. Perhaps some further extension of these studies as to the treatment of wire during the drawing operation may give a higher fatigue limit by a fortunate combination of formulas for the carbon content and the percentages of drawing. I merely suggest that as something to be studied.

A second application which is of considerable interest to power and light companies relates to the method of fastening guy wires, which is of economic interest. For high-strength strand the use of bolted clamps is almost universal. Clamps are expensive, and very often the cost of clamps in a typical guy will equal or even exceed the cost of the strand. If we can eliminate the clamps we can save money, but to do that we must serve the strand at terminuses and at insulators in the guy. Experience has been unfortunate in serving hard wire. It is brittle, and very often individual wires of the strand will break during the serving operation. Certain experiments performed a number of years ago indicated that the processing of the wire, the rate of drawing and the percentage of reduction had a vital effect on the success or failure of the serving operation.

Mr. Dartrey Lewis[3] (*by letter*).—I have been particularly interested in the three alternative methods of plotting the increase of tensile strength by wire drawing, as presented in Figs. 4, 5 and 6 of Mr. Godfrey's paper.

Figure 4, which plots tensile strength against reduction of area, gives the impression that the strength increases gradually during the early wire drawing, and then very rapidly in the later stages of wire drawing.

Figure 5, in which tensile strength is plotted against elongation, gives the impression that the strength increases very rapidly at first and then gradually tapers off.

In Fig. 6 the tensile strength is plotted against the logarithm of the strain. By this method of plotting, equal reductions of area per hole have equal horizontal spacing on the graph. It is believed that such a method of plotting is correct since, as every wire mill man knows, equal importance should be attached to, say, a 20 per cent reduction of area by wire drawing whether it occurs at the first, middle, or last hole. However, the formula for strain used by Mr. Godfrey is complicated and not very suitable for every-day use.

Equal spacing for equal reductions per hole can also be obtained by using a logarithmic scale on which is plotted the wire diameter. Mr. Godfrey's figures for the tensile strength of 0.67 per cent carbon steel have been replotted in the accompanying Fig. 1, using a logarithmic scale for wire diameters. At the bottom of the figure are shown the wire diameters for 10, 20, 30 and 40 per cent drafts. They illustrate that, with this type of plotting, equal reductions of area have equal horizontal spacings. Also plotted are the wire diameters corresponding to 10, 20, 30, etc., up to 95 per cent total reduction of area by wire drawing based on the original diameter. The logarithmic distortion of this scale should be noted.

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[2] Supervising Engineer, The Commonwealth & Southern Corp., Jackson, Mich.

[3] Chief Development Engineer, John A. Roebling's Sons Co., Trenton, N. J.

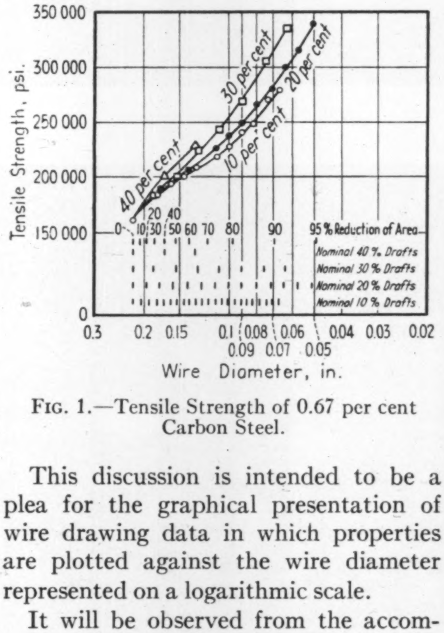

FIG. 1.—Tensile Strength of 0.67 per cent Carbon Steel.

This discussion is intended to be a plea for the graphical presentation of wire drawing data in which properties are plotted against the wire diameter represented on a logarithmic scale.

It will be observed from the accompanying Fig. 1 that the tensile strength increases more rapidly at the early and final stages of wire drawing than in the intermediate stages. However, up to 85 per cent total reduction of area by wire drawing no great error will result from assuming equal increments of tensile strength per hole (straight line graph). On such a basis, the following formula for tensile strength may be written:

$$S = K\,(\log D_1 - \log D_2)$$

where S = increase in tensile strength in pounds per square inch,
D_1 = initial diameter in inches,
D_2 = final diameter in inches, and
K = constant.

Applying this formula to Mr. Godfrey's figures for a 0.67 per cent carbon steel, the increase in tensile strength due to wire drawing is given by the following formulas:

10 per cent Drafting.

$$S = 210{,}000\,(\log D_1 - \log D_2)$$

20 per cent Drafting.

$$S = 234{,}000\,(\log D_1 - \log D_2)$$

30 per cent Drafting.

$$S = 295{,}000\,(\log D_1 - \log D_2)$$

MR. HOWARD J. GODFREY[4] (*author's closure, by letter*).—The author wishes to express his appreciation for the discussion of this paper. Mr. Winkler has emphasized the fact that there is no one optimum percentage of reduction which will produce the best physical properties for all kinds of steel wire. The author fully agrees with Mr. Winkler since the amount of wire drawing will be dependent on the desired physical properties for a given type of material and service.

The question of "size effect" in wire drawing is of extreme interest and might well be the subject of further research. There are certain fundamental changes that take place in steel as it is drawn into wire which are independent of the size of the wire. In the first instance wire drawing changes the original equiaxed structure to a tough fibrous structure. At the same time the plastic deformation work-hardens the steel and makes further deformation more difficult. Along with the fibering and work-hardening the original grains are fragmented and the

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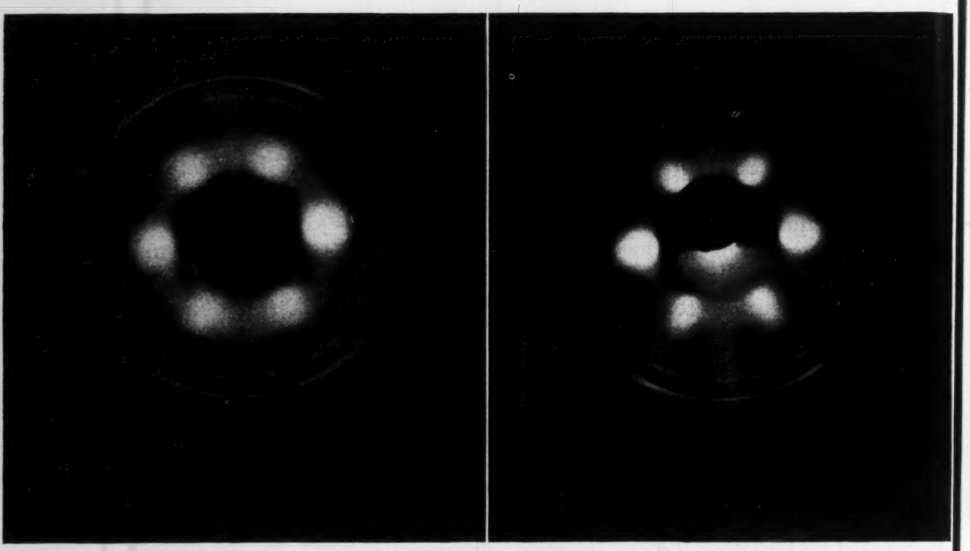
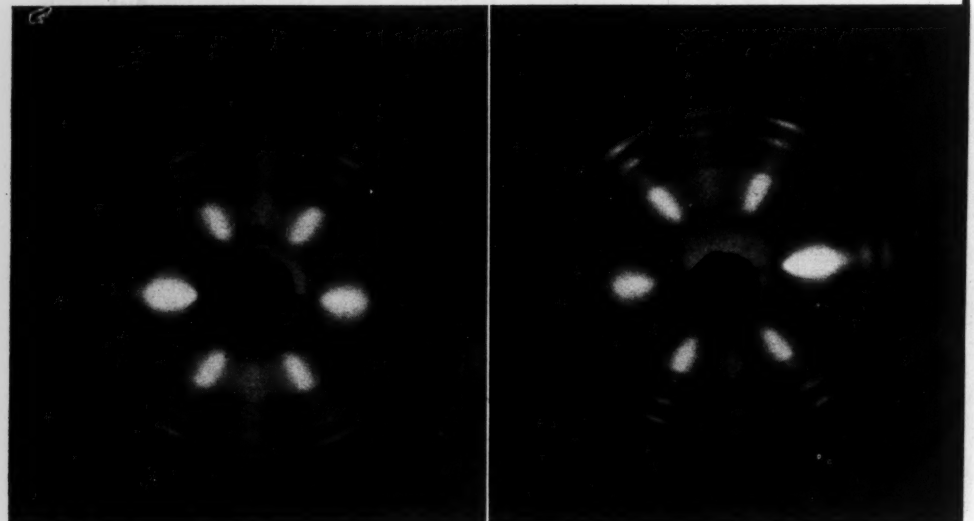
[4] Development Engineer, John A. Roebling's Sons Co., Trenton, N. J.



(*a*) 50.8 per cent reduction. (*b*) 75.5 per cent reduction.



(*c*) 88.2 per cent reduction. (*d*) 91.6 per cent reduction.

FIG. 2.—X-ray Diffraction Patterns of 0.67 per cent Carbon Steel Wire Drawn 30 per cent per Draft.

crystallites formed tend to become oriented in one specific direction. This preferred orientation increases with the wire drawing and can be measured by means of X-rays. The results of X-ray diffraction studies on the 0.67 per cent carbon wire drawn 30 per cent per draft are shown in the accompanying Fig. 2.

The examination was made on the material near the core of the specimens after first removing the outer layers with acid. The increase in the amount of preferred orientation represented by the concentration of the diffraction images into certain regions is very apparent as the amount of wire drawing increases. As a result of this preferred orientation the wire has definite directional properties.

Mr. Foote has discussed a practical problem in which two definite properties are desired in steel wire, that of high strength and toughness. There is no doubt that the method used in processing the wire will effect these physical properties. The bend test as described in this paper may be one means of determining the best type of wire for the service conditions discussed by Mr. Foote.

Mr Lewis has presented a method of plotting the physical properties which is very convenient, as it eliminates all computations for the true strain. The use of this method for denoting the amount of cold working would be of considerable aid in interpreting more logically the physical properties as affected by the amount of wire drawing.

The author wishes to acknowledge the work of W. F. Boyer, former student at Lehigh University, who carried out the X-ray studies under the direction of H. V. Anderson of the University.

COMPARATIVE QUALITY OF CONVERTER CAST STEEL

By C. E. Sims[1] and F. B. Dahle[1]

Synopsis

Because of a lack of adequate existing data, tests were run to determine the engineering properties of grade B converter cast steel in comparison with similar steels made by other melting processes. Steels made by acid open hearth, basic open hearth, acid electric, basic electric, Triplex (cupola-converter-electric) and side blow converter were used. Except for the lower phosphorus of the basic open-hearth steel, compositions were very similar. Mechanical properties at room temperature followed well-known effects of composition and inclusion type. Low-temperature notched-bar impact properties were found to be dependent upon deoxidation practice without relation to process of manufacture. The same was true with strain age-hardening effects. Standard fatigue and notched-bar fatigue results are in accord with tensile properties, rather than with notched-bar impact properties.

The authors conclude that the melting medium is of little moment in determining the properties of cast steel and that the only way to insure any desired property is to specify that property and test for it.

The all-out war effort in which we are now engaged demands the utmost use of all available and suitable agencies of production. An unprecedented demand for steel castings necessitates a greatly increased productive capacity. One method by which capacity may rapidly be expanded is the cupola-converter combination. This method of steel production has the further advantage that many of the cupolas are already available. Numerous gray iron foundries have been forced to discontinue manufacture of their normal products, and this has made available, not only cupolas, but plants fully equipped with sand-handling and molding facilities and crews of experienced molders. While not experienced in steel casting, they can learn the proper technique faster than green men.

The possibilities of converter steel may have been overlooked because its use was prohibited by certain specifications. Despite the great need for production and more production, there is no logic in using materials not known to be suitable for a given purpose. Both the Army and Navy Ordnance Departments have used meticulous care to avoid falling into this error and have specified materials that they were confident would maintain the highest quality in ordnance.

In the case of cast steel, there were no adequate technical data with which to make an impartial appraisal of the quality of converter steel as compared to cast steel made by other processes and proven by actual use to be suitable. Although practically all cast converter steel is made in the side-blow converter, it was put in the general classification of converter or Bessemer steel, which for many years has been considered, not always with justice considering modern practice, to be inferior in quality to open-hearth or electric steel. The present paper supplies data by which the comparative quality of cast converter steel may be judged.

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[1] Supervising Metallurgist, and Assistant Supervisor, respectively, Battelle Memorial Institute, Columbus, Ohio.

The electric furnace was introduced to the foundries of the United States in 1909 at a time when crucible steel was relatively expensive and the converter was almost the only producing medium open to the small jobbing foundry. The converter reached peak production in 1918 with 180,000 tons, but by 1919 the electric furnace had passed it. By 1930 the converter had fallen to a production of 16,500 tons.

Hamilton (**1**)[2] has clearly stated the conditions that led to the adoption of the electric furnace, but suffice it to say that they were mainly economic and not quality considerations. Deoxidation practice was not well understood and, as a result, there was a prevalent belief that the electric furnace could make the highest quality of steel because it could maintain a reducing atmosphere throughout the melt. This view has been modified, and today steel metallurgists in general regard it as a fact that the best quality cast steel is made when a thorough oxidation is part of the melting program.

Some converter shops have remained in continuous operation, and these have experienced no trouble in meeting ordinary commercial specifications. In England, a land of relatively small foundries, the converter was reported (**2**) in 1936 to be still the most widely used steel foundry melting medium. Converter cast steel is well known for its high fluidity and relative freedom from pinhole porosity.

The bad name acquired by converter steel must have been earned, and early converter steel was a relatively impure and unrefined steel. Most Bessemer steel was, and still is, made by blowing hot metal direct from the blast furnace. Side blow converters used cupola melted metal, and the charge to the cupola consisted mostly of pig iron with a small amount of scrap steel and scrap cast iron. Only acid converters have been used in the United States, and in these there is no phosphorus or sulfur removal. Indeed, these impurities were increased because of oxidation of iron. Because of inadequate controls, unusually high nitrogen contents were sometimes acquired. Thus converter steel was conceded to be impure, with high contents of phosphorus, sulfur, and nitrogen, and to be inferior in some mechanical properties.

Today conditions are changed. The modern hot-blast cupola can handle an all-steel charge, and a charge low in phosphorus and sulfur can be selected. Some sulfur is picked up from the cupola coke, but the soda ash treatment of molten cast iron for sulfur removal has been developed to such a state of efficiency that sulfur specifications can readily be met with converter steel. If a high-phosphorus charge has been used and the phosphorus content of the blown metal is too high, it can be brought within bounds by a ladle treatment with a basic, oxidizing slag. Such treatments have been only recently developed but are proving successful in operation. Control of the metal composition charged to the converter and control of operation, such as is provided by the photoelectric cell, keep nitrogen content of the blown metal to a minimum.

Now that side-blow converter cast

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[2] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 550.

steel can be made to the same chemical composition as acid open hearth or acid electric cast steel, the present question is, does it nevertheless differ essentially in mechanical properties? It seemed that an appraisal of these properties was in order.

The investigation was sponsored by the Whiting Corp. for the purpose of determining the facts, and contemplated as a follow-through a study of means to correct any deficiencies that might be found. All of the steels used were from regularly produced commercial heats. It is recognized that steel castings are products of three factors: (1) the inherent engineering properties of the steel that is used, (2) the mechanical integrity of the casting, and (3) the heat treatment.

The mechanical integrity of a casting is largely a matter of foundry technique, and no attempt was made here to determine whether, as is often claimed, converter steel has superior castability. The heat treatment was a constant, and the investigation was concerned only with the comparative engineering properties of these steels, under standardized conditions.

MATERIALS USED

Specimens of cast steel for testing were solicited from various commercial foundries. These specimens were in the form of coupons cut from keel blocks of the design shown in Fig. 1. All specimens were from the same design of keel block, except two heats (9551 and 9567, Table I), which differed only by being slightly greater in cross-section. The processes represented by the specimens are as follows: acid open hearth, basic open hearth, acid electric, basic electric, "Triplex" (cupola-converter-electric), and side-blow converter.

An effort was made to obtain at least two heats of each type from each contributing shop and to have two shops represented by each process. Triplex heats were obtained from only one shop and only one basic electric steel was secured. The request was made that all of these steels represent the current standard practice in use at any particular shop. The only exception to this was in converter heats 4 and C in which it was requested that titanium be used in addition to the regular deoxidizers and in converter heat D in which an addition of 2 lb. of aluminum per ton was requested.

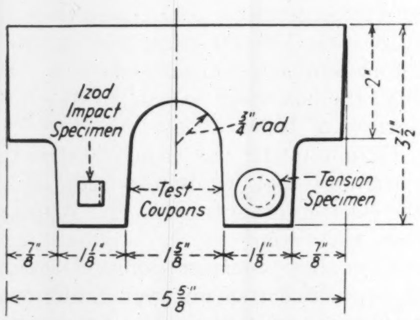


FIG. 1.—Cross-Section of Keel Test Block Used for Specimen Castings.

In view of the prevalent use of aluminum in cast steels, the normal expectation would have furnished some open-hearth or electric steels deoxidized with aluminum. Such was unfortunately not the case and, in view of the trend of the results, two acid electric heats deoxidized with 2 lb. of aluminum per ton were later especially requested. Only one of these, heat 1163, was acquired in time for inclusion. This came from the same shop as heats 715 and 724.

Heat Treatment:

All coupons were received in the green or as-cast condition and all heats were given the same heat treatment. This consisted of heating the coupons to

1650 F., holding for 2 hr., and cooling separately in still air. One set of tension bars was machined and tested within three days of the normalizing treatment.

TABLE I.—CHEMICAL ANALYSES OF GRADE "B" CAST STEELS.

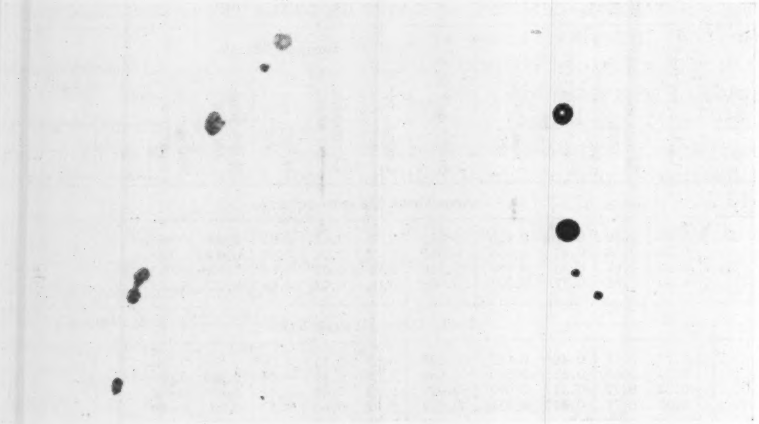
Steel	Carbon, per cent	Manganese, per cent	Silicon, per cent	Phosphorus, per cent	Sulfur, per cent	Residual Metals					Special Deoxidizers Added to Ladle, lb. per ton			
						Copper, per cent	Chromium, per cent	Nickel, per cent	Molybdenum, per cent	Nitrogen, per cent	Al	Fe Ti	Ca Si	Ca Si Mn
ACID OPEN HEARTH STEELS														
493	0.21	0.69	0.42	0.030	0.035	0.11	0.05	0.01	0.006	0.006	...	2	3	...
509	0.24	0.66	0.47	0.036	0.035	0.12	0.04	0.01	0.006	0.005	...	2	3	...
F-1	0.33	0.67	0.36	0.036	0.036	0.05	0.02	0.04	0.060	0.005	...	...	...	...
F-2	0.30	0.85	0.37	0.035	0.033	0.04	0.02	0.04	0.051	0.006	...	...	...	...
BASIC OPEN HEARTH STEELS														
9551	0.27	0.58	0.46	0.013	0.025	0.15	0.14	0.06	0.016	0.005	...	...	...	...
9567	0.27	0.68	0.45	0.019	0.034	0.10	0.12	0.05	0.012	0.005	...	...	...	...
O-1	0.32	0.72	0.34	0.017	0.027	0.12	0.05	0.15	0.04	0.004	...	...	...	...
O-2	0.30	0.77	0.34	0.016	0.029	0.12	0.06	0.17	0.03	0.004	...	...	...	...
ACID ELECTRIC STEELS														
715	0.27	0.80	0.45	0.027	0.034	0.06	0.05	0.03	0.013	0.008	...	3	...	3
724	0.26	0.72	0.39	0.026	0.036	0.08	0.03	0.06	0.009	0.008	...	3	...	3
1009	0.26	0.68	0.40	0.034	0.038	0.11	0.06	0.18	0.038	0.010	...	4	...	1.5
1025	0.24	0.66	0.39	0.035	0.040	0.11	0.06	0.16	0.034	0.009	...	4	...	1.5
1163	0.26	0.68	0.49	0.036	0.037	...	...	...	...	0.009	2	...	...	...
BASIC ELECTRIC STEELS														
E-1	0.34	0.88	0.28	0.033	0.035	0.12	0.19	0.43	0.06	0.007	0.7	...	...	...
CONVERTER-ACID ELECTRIC STEELS (TRIPLEX)														
H-30	0.30	0.73	0.40	0.029	0.035	0.36	0.04	0.08	0.056	0.011	2[a]	...	2	...
H-34	0.34	0.78	0.40	0.033	0.036	0.39	0.04	0.06	0.062	0.011	2[a]	...	2	...
H-38	0.33	0.58	0.31	0.023	0.041	0.38	0.03	0.08	0.005	0.012	2[a]	...	2	...
H-42	0.35	0.73	0.35	0.059	0.047	0.43	0.03	0.10	0.004	0.010	2[a]	...	2	...
CONVERTER STEELS														
1	0.26	0.77	0.55	0.034	0.041	0.19	0.04	0.09	0.008	0.008	2	...	...	...
2	0.26	0.66	0.38	0.032	0.027	0.09	0.04	0.06	0.006	0.009	2	...	...	...
3	0.27	0.65	0.37	0.038	0.026	0.09	0.05	0.06	0.007	0.009	2	...	...	...
4	0.26	0.50	0.32	0.033	0.028	0.14	0.03	0.05	0.007	0.009	2	2	...	...
A	0.29	0.50	0.33	0.040	0.031	0.09	0.02	0.085	0.008	0.006	1	...	...	...
B	0.33	0.85	0.43	0.043	0.035	0.09	0.02	0.072	0.018	0.008	1	...	...	...
C	0.33	0.64	0.39	0.049	0.038	0.10	0.03	0.079	0.018	0.007	1	1.7	...	...
D	0.29	0.73	0.36	0.044	0.035	0.09	0.03	0.077	0.014	0.007	2.25	...	...	...

[a] Aluminum added to electric furnace in these heats.

All the rest of the coupons were annealed at 750 F. for 15 hr. This annealing temperature was chosen because it is known to eradicate hydrogen embrittlement without affecting the tensile strength of a normalized medium-carbon steel. Aging at normal temperatures for a longer period will accomplish the same result. Any improvement in ductility produced by this low-temperature anneal is regarded, therefore, as a qualitative evidence of hydrogen embrittlement in the normalized bars.

Composition of Steels:

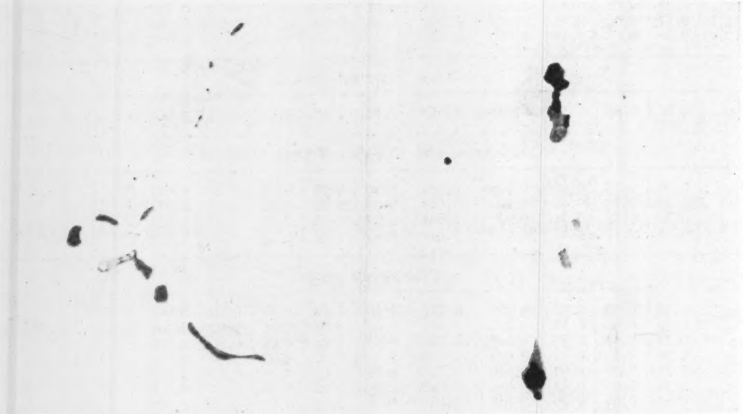
In most instances, plant analyses were furnished with the steel specimens, but all steels were analyzed at Battelle and, in addition to the common elements, copper, chromium, nickel, molybdenum, and nitrogen were determined. In those few cases where the plant analyses and Battelle determinations did not agree, an umpire analysis was made by a third laboratory.



Sulfides

Silicates

(*a*) Type I inclusions.



(*b*) Type II inclusions.

(*c*) Type III inclusions.

FIG. 2.—Types of Inclusions Found in Cast Steels, Unetched (× 500).

The compositions are shown in Table I. It will be noted that although the carbon varies from 0.21 to 0.35 per cent, the manganese from 0.50 to 0.88 per cent, and the silicon from 0.28 to 0.55 per cent, they all come in the grade B classification. Some question might be raised in regard to the chromium and nickel content of the basic electric furnace steel E-1 and the copper content of the Triplex steels because these residuals undoubtedly affect the properties as will be pointed out later.

As might be expected, the basic open-hearth steels are lower in phosphorus than any of the others, while the converter steels as a class are slightly higher in phosphorus than the acid open-hearth and electric steels. The Triplex steels were given a ladle dephosphorization treatment which was effective except in heat H-42 where converter slag got into the ladle and made the dephosphorizing slag too acid.

The basic open hearth also has a slight advantage in sulfur content, but the desulfurizing treatment of the cupola metal has put the converter steels on a par with the acid electric and acid open hearth in respect to this impurity.

TABLE II.—MECHANICAL PROPERTIES OF GRADE "B" CAST STEELS.

NOTE.—Tensile values and Charpy impact values are averages of duplicate tests. Izod values are averages of three tests.

Steel	Yield Strength, psi.		Tensile Strength, psi.		Elongation, per cent		Reduction of Area, per cent		Brinell Hardness As Normalized and Drawn	Impact Strength As Normalized and Drawn, ft-lb.		A.S.T.M. Grain Size Normalized and Drawn	Type of Sulfide Inclusions
	As Normalized	As Normalized and Drawn	As Normalized	As Normalized and Drawn	As Normalized	As Normalized and Drawn	As Normalized	As Normalized and Drawn		Izod	Charpy		
ACID OPEN HEARTH STEELS													
493	49 000	46 500	74 500	74 500	33.5	32.0	57.0	55.5	147	34	25	7 to 8	I
509	46 000	43 000	75 000	75 000	33.0	34.0	57.0	56.5	142	32	24	7 to 8	I
F-1	46 500	46 000	84 000	83 000	28.0	29.0	47.0	46.5	162	22	18	6 to 7	I
F-2	49 000	47 500	86 500	87 000	29.5	29.0	49.5	49.5	169	22	18	6 to 7	I
BASIC OPEN HEARTH STEELS													
9551	45 000	45 500	74 000	75 500	29.0	33.0	38.0	56.5	144	40	27	6 to 7	I
9567	46 000	46 000	78 000	79 500	31.5	32.5	56.5	57.0	150	36	26	7	I
O-1	48 500	46 500	82 500	81 500	31.0	31.5	52.5	52.5	162	34	22.5	7	I
O-2	47 500	48 000	80 000	80 500	32.0	33.0	53.5	55.5	157	29	24	6 to 7	I
ACID ELECTRIC STEELS													
715	56 000	48 000	83 000	81 000	28.5	28.5	49.0	49.5	165	28	20	8	I
724	52 500	47 500	80 000	80 000	30.5	30.0	54.0	54.0	157	29	23	8	I
1009	50 500	49 000	82 500	82 000	29.0	30.0	53.0	52.0	162	25	20	7	I
1025	47 000	47 000	78 500	78 500	32.0	30.5	53.0	54.5	155	27	21.5	7	I
1163	47 000	46 000	75 000	75 000	27.0	29.0	40.0	45.0	142	...	22	7 to 8	III
BASIC ELECTRIC STEELS													
E-1	59 000	60 500	92 000	93 500	20.0	23.0	26.5	33.0	188	19	12	7 to 8	II
CONVERTER-ACID ELECTRIC STEELS (TRIPLEX)													
H-30	58 500	53 500	84 500	87 000	28.0	26.0	44.5	35.5	170	34	19	8	III
H-34	58 000	55 500	87 500	87 500	25.5	24.5	35.0	37.0	174	33	18.5	8	III
H-38	47 500	47 500	81 000	81 000	20.0	24.0	29.0	33.5	157	27	15	6 to 8	III
H-42	61 000	55 500	90 000	91 500	23.0	23.5	32.0	30.0	176	21	13	8+	III
CONVERTER STEELS													
1	53 000	53 500	80 500	81 000	29.5	31.0	45.0	49.5	155	38	21	8	III
2	47 500	46 000	74 500	75 000	28.0	33.0	42.5	55.5	146	44	28	8	III
3	48 500	48 000	76 500	77 500	27.0	31.0	44.5	53.0	152	42	26	7 to 8	III
4	47 000	47 000	72 500	73 500	29.0	31.0	47.0	50.0	143	43	25.5	8	III
A	46 000	44 500	77 000	76 500	28.0	31.5	40.5	52.0	148	28	23	7	III
B	59 500	58 500	87 500	88 000	19.0	24.0	22.5	34.5	172	26	13	8	II
C	55 500	55 000	85 500	85 000	22.5	23.5	32.0	31.0	168	30	13	8	II
D	52 000	50 000	81 000	80 000	27.0	30.0	47.0	48.5	157	39	23.5	7 to 8	III

Nitrogen contents were determined by

the Cunningham distillation method which is standard for wet methods, but the accuracy of the reported results is not considered greater than ±0.002 per cent. Certain trends, nevertheless, are apparent. The Triplex steels have the highest content with an average of about 0.011 per cent; the straight converter steels and acid electric steels are in the same range of 0.006 to 0.010 per cent, while the open-hearth steels have a still lower range of 0.004 to 0.006 per cent.

TABLE III.—LOW TEMPERATURE NOTCHED-BAR IMPACT PROPERTIES OF GRADE "B" STEELS.

Steel	Type of Sulfide Inclusions	Single-Width Charpy (Keyhole Notch) Impact Strength, ft-lb.															
		212 F.		70 F.		32 F.		0 F.		−20 F.		−40 F.		−60 F.		−80 F.	
		Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.
ACID OPEN HEARTH STEELS																	
493	I	30.5	30.0	27.5	23.0	22.5	21.5	21.0	20.0	19.5 *	3.5	17.0	2.5	2.0	2.0	....	....
509	I	32.5	32.5	24.5	24.0	18.5	18.5	22.5 *	5.5	20.0	4.0	17.0	2.0	2.0	2.0	....	....
F-1	I	22.0	21.5	19.5	16.5	13.5	12.5	14.5 *	7.0	8.0	2.0	3.0	1.0	1.5	1.5	....	....
F-2	I	....	....	18.0	17.5	16.0	14.5	14.0	13.0	12.0 *	3.0	2.0	1.5	1.0	1.0	....	....
BASIC OPEN HEARTH STEELS																	
9551	I	34.5	32.0	27.5	27.0	23.0	22.0	23.0	22.0	20.5	20.0	16.5 *	3.5	18.0	3.0	....	....
9567	I	34.5	34.0	27.0	25.0	22.0	22.0	21.0	21.0	21.5	19.5	18.0 *	2.5	10.0	2.0	....	....
O-1	I	....	....	24.5	20.5	18.0	12.0	16.0	16.0	17.0 *	4.5	5.0	2.5	2.0	2.0	....	....
O-2	I	28.5	28.5	25.0	22.5	21.0	20.5	15.5	14.0	13.0 *	3.5	3.0	2.5	....	....	....	....
ACID ELECTRIC STEELS																	
715	I	29.0	26.5	20.0	19.5	21.5	18.0	14.5 *	4.0	19.0	2.0	2.0	2.0	....	....	....	....
724	I	27.0	26.0	24.0	22.0	19.0	16.0	18.5	18.0	15.0 *	3.5	2.0	2.0	....	....	....	....
1009	I	25.0	24.5	21.5	18.0	14.0	13.0	15.0 *	5.0	10.0	2.5	7.5	1.0	1.0	1.0	....	....
1025	I	27.0	26.0	22.5	20.5	15.5	13.5	19.0	17.5	16.0	14.0	9.0 *	1.0	1.0	1.0	....	....
1163	III	23.0	19.5	23.5	21.0	20.0	19.0	17.5	16.5	15.5	15.0	12.5	11.5	13.5	8.5	9.5 *	2.0
BASIC ELECTRIC STEELS																	
E-1	II	15.0	13.5	12.5	12.0	10.0	10.0	7.5	7.0	6.5	5.0	6.5	6.0	3.5	2.5	....	....
CONVERTER-ACID ELECTRIC STEELS (TRIPLEX)																	
H-30	III	22.0	20.0	19.5	18.5	18.5	17.0	12.5	11.5	10.0	9.5	10.5	9.0	15.5	11.0	....	....
H-34	III	....	....	19.0	18.0	13.0	11.5	16.0	11.0	10.5	9.0	13.5	8.5	9.0	8.5	....	....
H-38	III	18.0	14.0	16.0	14.5	13.0	12.0	10.5	10.0	11.5	10.0	10.5	10.0	9.0 *	5.0	6.5	5.5
H-42	III	....	....	14.0	12.5	10.0	9.0	9.5	7.0	12.5	12.0	14.0	6.5	5.0 *	4.0	3.0	2.0
CONVERTER STEELS																	
1	III	24.0	21.0	22.5	20.0	20.0	18.0	14.5	14.0	17.0	16.0	14.5	12.5	15.5	15.0	12.0	12.0
2	III	32.0	30.0	27.5	27.0	25.0	24.0	23.0	23.0	21.0	20.5	19.5	17.5	18.0	16.0	14.5	14.5
3	III	30.0	30.0	26.0	25.5	25.0	23.0	24.0	20.5	20.0	18.5	18.5	13.5	18.0	17.5	17.0	14.5
4	III	30.0	29.5	26.0	25.0	22.0	18.5	21.5	21.0	19.5	17.5	16.0	11.0	17.0	15.5	15.5 *	9.0
A	III	22.0	22.0	24.0	21.5	....	....	16.0	14.5	16.5 *	9.5	11.0	5.0	10.0	8.5	5.5	3.5
B	II	....	....	13.5	12.0	10.0	10.0	9.5	9.0	7.5	7.0	5.0 *	4.5	7.0	5.5	2.5	2.0
C	II	13.0	13.0	13.0	12.5	10.0	10.0	14.0	7.5	10.0 *	4.5	3.5	2.5	4.5	2.0	2.0	1.5
D	III	....	....	23.5	23.5	18.0	15.0	16.0	11.5	12.5	11.5	11.5	11.5	13.0	9.5	7.5 *	5.0

* Asterisk placed in data at temperature where low or erratic results were first encountered.

Microstructure:

Microscopic examinations were made to determine the nature of the inclusions and the grain size, inasmuch as both are known to affect the mechanical properties. Except that the high sulfur steels had more sulfides than those low in sulfur, there was no notable difference in cleanliness.

The type of inclusion was found to coincide closely with the deoxidation practice and particularly with the use of aluminum, as described by Sims and

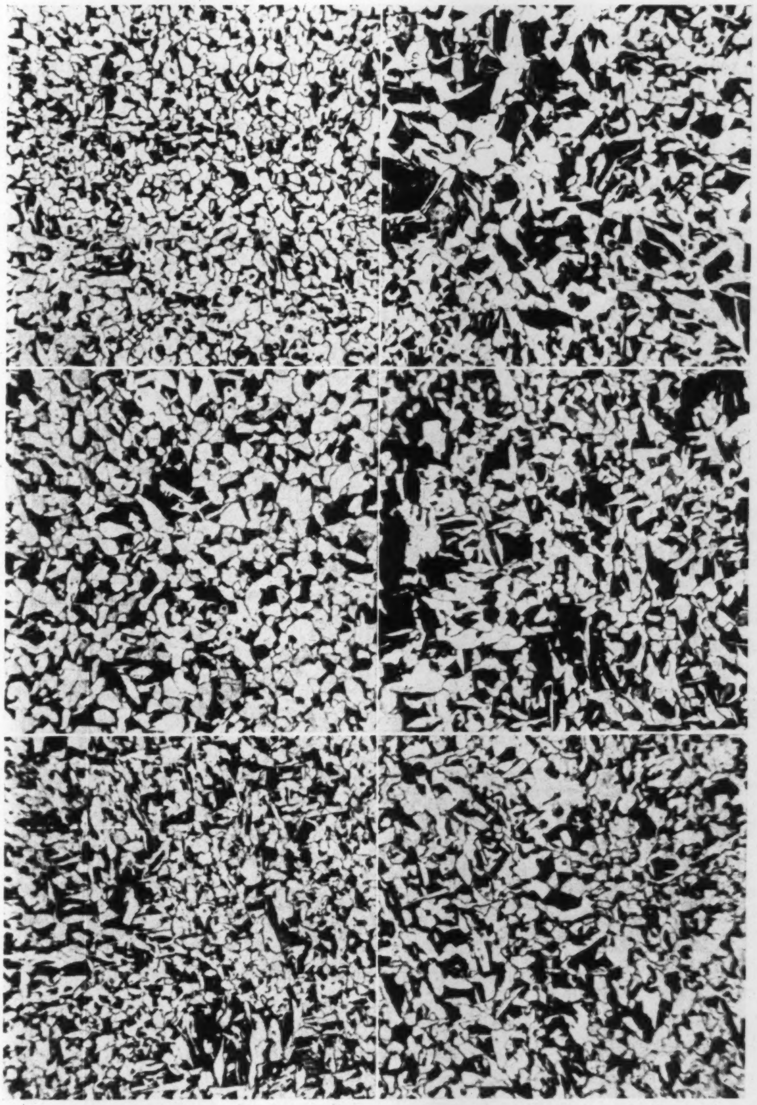


FIG. 3.—Grain Structures of Cast Steels, Etched in Nital (× 100).

Upper micrographs—acid open hearth steels 493 (left) and F-1 (right).
Middle micrographs—basic open hearth steels 9551 (left) and 0-1 (right).
Lower micrographs—acid electric steels 715 (left) and 1009 (right).

Dahle (3). When no aluminum was used, regardless of whether calcium or titanium were used, the inclusions consisted of globular sulfides and silicates as shown in Fig. 2(*a*). These are designated type I and steels with this type are inclined to have good ductility.

When additions of aluminum too small to leave an appreciable excess are used, the silicates disappear and the sulfides assume the form of an eutectic as shown in Fig. 2(*b*). Such chain or film type sulfides, strung along the primary grain boundaries, act as zones of weakness and produce poor ductility. This was described by Sims and Lillieqvist (4). These inclusions are designated as type II.

If aluminum is used in sufficient amount to leave an appreciable excess, the sulfides are larger, farther apart, and are irregular in shape or are crystalline. Many duplex inclusions appear. These are type III inclusions and an example is shown in Fig. 2(*c*). Steels with these inclusions have good ductility but not quite so good as with the type I inclusions. The type of inclusions is shown in Tables II and III.

The grain size listed in these tables is the grain size of the steels as normalized from 1650 F., the condition in which

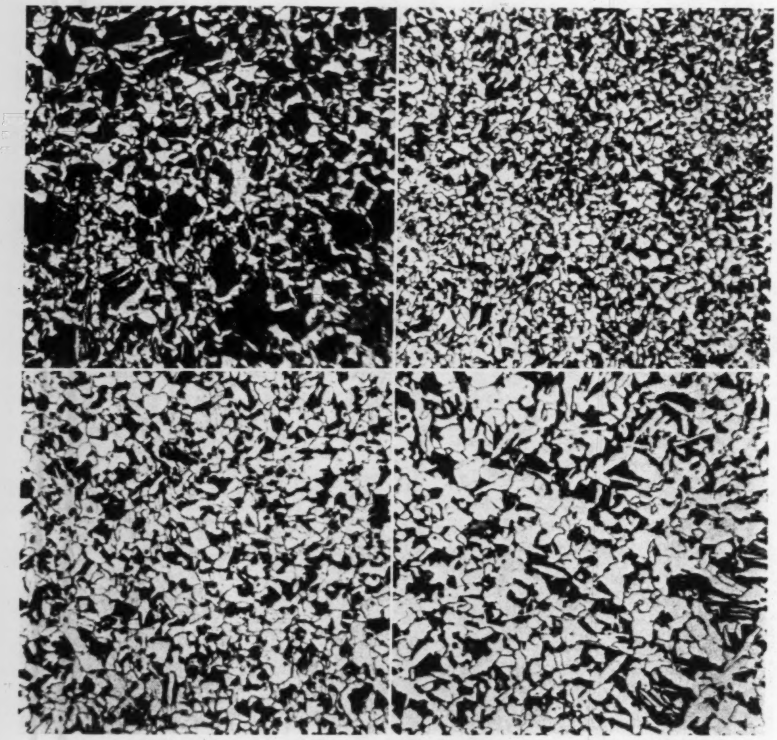


FIG. 4.—Grain Structures in Cast Steels, Etched in Nital (× 100).

Upper left—basic electric steel E-1.
Upper right—Triplex steel H-30.
Lower left—converter steel 1.
Lower right—converter steel A.

they were tested. Although the grain size does not vary over a wide range, it does show the aluminum deoxidized steels to have the smallest grain size, with the titanium deoxidized steels showing an advantage over those with no special deoxidizer. This is illustrated in Figs. 3 and 4 where examples of each series of steels are shown.

Mechanical Properties

Room-Temperature Tests:

The data in Table II, covering tension, hardness, and notched bar-impact tests, give a general comparison of the properties most commonly determined for these steels. The yield and tensile strengths shown are slightly higher than those ordinarily obtained commercially for similar compositions because the individually handled laboratory bars cooled somewhat faster. The yield and tensile strengths, however, follow the compositions very closely. High carbon or manganese content, or a combination of high carbon and high manganese, produce high strength. The chromium and nickel content of heat E-1 has increased the strength of this steel, whereas the copper in the Triplex steels has a negligible effect.

The draw or low-temperature anneal caused a slight average drop in the yield strength but had no effect on the tensile strength.

Other things being equal, the ductility tends to vary with the composition. Carbon and sulfur are the most potent elements and the ductility decreases as either or both of these elements increase. Superimposed on these effects, however, is the strong effect of inclusion type and it will be observed that all of the steels with type I inclusions have very good ductility.

The three steels with type II inclusions, E-1, B, and C, all have poor ductility, although this can be accounted for in part by the high carbon contents. With the exception of the Triplex steels, all of the steels with type III inclusions have good ductility but average a little lower than those with type I. The ductility of the Triplex steels is somewhat lower than can be accounted for by composition, but some of the inclusions in these heats are close to type II.

Hydrogen:

Hydrogen embrittlement, as indicated by the improvement of ductility produced by the draw, is present in the basic open-hearth heat 9551, acid electric heat 1163, basic electric heat E-1, and the converter steels 1, 2, 3, 4, A, and B. This indicates a greater prevalence in the converter steels, although some trouble with hydrogen is not uncommon in the other steels. There is some indication that present practices tend to minimize this trouble.

Brinell Hardness:

Brinell hardness numbers were taken on sections cut from the center of the coupons and, as usual, follow the tensile strengths very closely.

Notched-Bar Impact:

Notched-bar impact tests were made both on V-notch Izod specimens and keyhole-notch Charpy specimens. The Izod values, although higher, follow the same trends as the Charpy figures and both, in general, bear a close correlation to the ductility. Comparing steels with the same ductility, however, it is apparent that the aluminum deoxidized steels have the highest average impact ratings, whereas those treated with titanium show no advantage over those without a special deoxidizer. It so happens that most of the aluminum deoxidized steels are converter steels, but because this effect of aluminum is well known, no significance is given to the method of production.

All of the properties in Table II, therefore, seem quite orthodox for grade B steel and can be accounted for by composition and deoxidation practice.

Low-Temperature Notched-Bar Impact Tests:

One of the properties in which converter steel has been alleged to be especially vulnerable is notch sensitivity at low temperatures. However, data are on the record (5) for properly aluminum-killed Bessemer pipe (0.15 per cent carbon) that gave Charpy values of 50 ft-lb. at room temperature, 34 at −25 F., and 21 at −50 F. Large tonnages of such pipe have been in successful commercial use over a period of years. While this behavior of wrought steel leads to the expectation of similar behavior of cast steel, direct evidence was lacking.

Considerable attention was given, therefore, to low-temperature notched-bar impact tests in the present study. It is well known that such tests differentiate sharply between steels properly and improperly deoxidized, whereas the static tensile properties at low temperatures do not. All the impact tests at low temperatures were made with standard keyhole-notch Charpy bars, single and double width.

The specimens were cooled by immersion in a bath of acetone and dry ice and were broken within 3 sec. after removal from the bath. During this interval, they would warm up from 2 to 4 F. and, to compensate, the bath was held to a temperature 3 F. below the desired breaking temperature. Each bar was held 15 min. at temperature while the bath was agitated, before testing.

In all cases, the tests were made in duplicate but further check tests were made when it seemed to be desirable. Check tests were made primarily where the original duplicate tests gave erratic results. In Table III, where the data for single width specimens are recorded, only the maximum and minimum values are listed, regardless of the number of tests made.

It will be observed from this table that the impact values gradually decrease with drop in temperature until suddenly they become very erratic or drop to a much lower figure. It is this drop-off, where the steels change from a relatively tough to a brittle material, that is important to determine. It is obvious, of course, where duplicate tests give such values as 17 and 2 ft-lb., that chance might just as well have given either two high values or two low values. Too much importance, therefore, must not be placed on individual figures; trends are of greater moment.

Among the acid open-hearth heats, the first heat begins to show erratic values at −20 F., the second and third at 0 F., and the fourth at −20 F. At −40 F. three show extremely low values.

The basic open-hearth heats held up better and the first two show no drop-off until −40 F., while the next two drop off sharply at −20 F. The better properties of these steels may be attributed tentatively to the relatively low phosphorus content. These steels had no special deoxidation.

Erratic results were obtained at 0 F. for the first and third acid electric steels, at −20 F. for the second, while the fourth failed at −40 F. The fifth heat of this series (1163), however, exhibits no erratic behavior but only a gradual reduction in impact value down to −80 F., where a sharp drop was obtained. It should be noted here that the first four steels were treated with

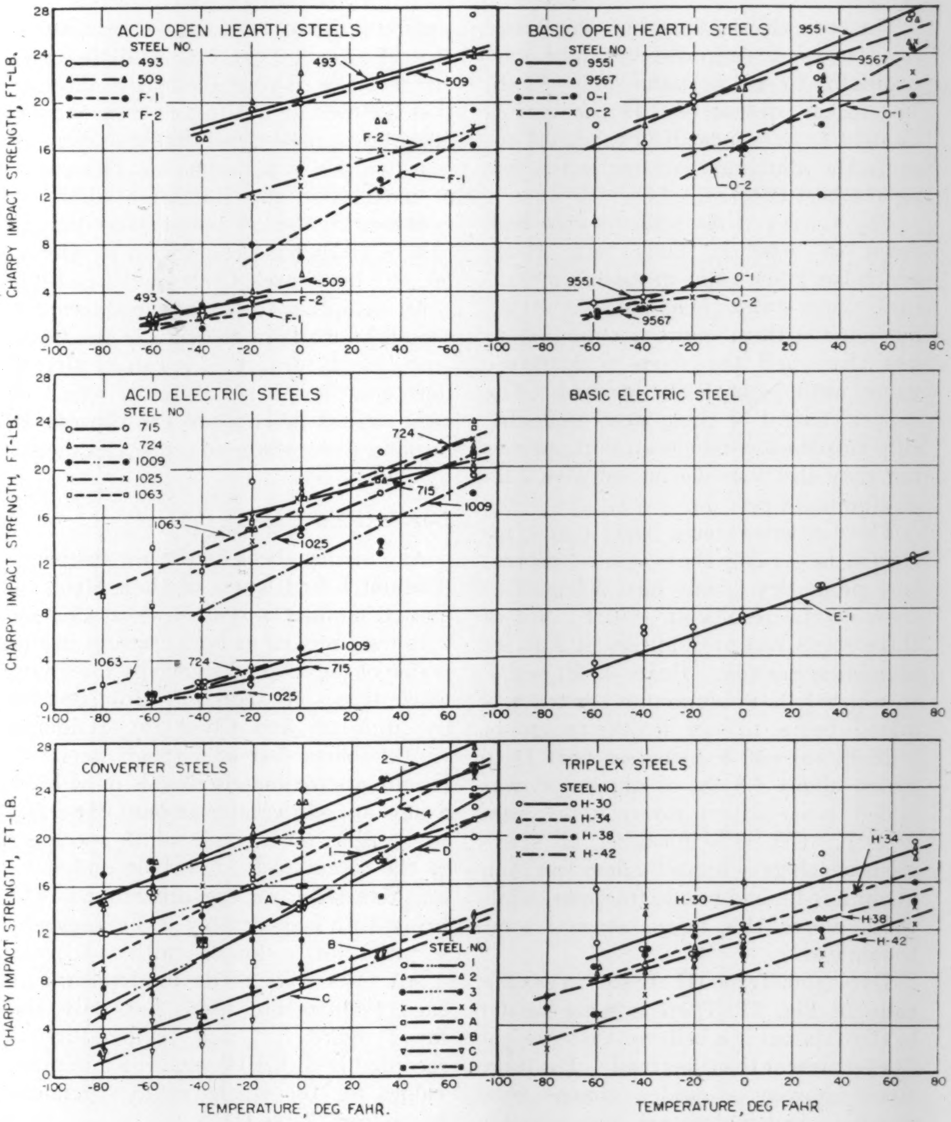


FIG. 5.—Low-Temperature Notched-Bar Impact Strength of Cast Steels.

(Curves plotted from values in Table III) titanium and calcium, whereas the last was deoxidized with 2 lb. of aluminum per ton.

The basic electric heat, starting rather low, shows no inconsistency and falls gradually to a low value at −60 F. The nickel content of 0.43 per cent is thought to be of benefit in this steel because the aluminum content is too low for the best effect.

The Triplex steels held up very well down to −60 F. Only heat H-42, which has a low value at room temperature, shows erratic behavior at −40 F., but none of them shows a reduction to less than half the room temperature value until −60 F. is reached. The copper content of these steels undoubtedly aids the low-temperature properties but they also were deoxidized with 2 lb. of aluminum per ton.

The converter steels, heats 1 to 4, inclusive, have easily the best low-temperature properties. Only heat 4 begins to show erratic results at −80 F. All of these steels had an addition of 2 lb. of aluminum per ton. Heats A, B, and C to which 1 lb. of aluminum per ton was added begin to fail in the range of −20 F. to −40 F., whereas heat D to which about 1.5 lb. of aluminum was added is consistent down to at least −60 F. Generally speaking, all specimens which gave impact values less than about 5 ft-lb. had brittle fractures, while those above this figure showed some toughness.

Data from Table III are shown graphically in Fig. 5. There is some scatter in the data but it is believed that straight lines represent the true trend. For those steels showing a sudden change from tough to brittle fracture two separate curves are drawn, because in most instances, there are no intermediate data. Where the upper and lower curves overlap, however, there is apt to be some erratic results.

Superficially, these low-temperature notched-bar impact values seem to show a distinct advantage for converter (including Triplex) steel, but the evidence of acid electric heat 1163 and the converter steels B and C clearly indicate that the melting medium is of secondary importance at best and that the deoxidation practice is paramount. The value of deoxidation and aluminum additions in enhancing the low-temperature toughness of steels has been shown by Herty (6), and by Kinzel, Crafts and Egan (7).

An exception to the above statement should be made in the case of the basic open-hearth furnace, if it can be shown that low phosphorus content, which is more or less inherent to the process, is responsible for the good properties shown by these steels.

Double Width Charpy Bars:

As a further check on the low-temperature notch brittleness of these steels, a limited number of double width Charpy bars were broken at temperatures in the region of the drop-off shown by the single width bars. As shown by Hoyt (8) and by McAdam and Clyne (9), a double width Charpy bar of a tough steel will require approximately double or at least a substantially greater amount of energy to break as for a single width specimen of the same steel. A brittle or notch-sensitive steel, on the other hand, will break with considerably less energy absorption for a double-width specimen.

All tests were made in duplicate at each temperature used, but only the lower figure for each temperature is recorded in Table IV with the minimum values for the single width specimens taken from Table III.

It might be expected that these results would show a different trend or be more erratic, yet they present substantially the same picture. For convenience, asterisks are placed between results at the highest temperature where the double width value is not substantially greater than the single width value or where both figures are very low. The position of these asterisks corresponds very closely to where the notch brittleness was first manifested by the single width results and the advantages of proper deoxidation is demonstrated just as clearly. In general, the first signs of brittleness are observed at a slightly higher temperature with the double-width specimens, but in a few cases, this is reversed.

TABLE IV.—COMPARISON OF LOW-TEMPERATURE NOTCHED-BAR IMPACT STRENGTHS FOR SINGLE AND DOUBLE WIDTH SPECIMENS.

NOTE.—Minimum values of two or more tests given in table. Asterisk placed between values where double width specimens first do not show substantially greater values than single width specimens or where both values are very low.

Steel	Charpy (Keyhole Notch) Impact Strength, ft-lb.											
	Tests at 32 F.		Tests at 0 F.		Tests at −20F.		Tests at −40 F.		Tests at −60 F.		Tests at −80 F.	
	Single Width	Double Width	Single Width	Double Width	Single Width	Double Width	Single Width	Double Width	Single Width	Double Width	Single Width	Double Width
Acid Open Hearth Steels												
493	21.5	45	20	37.5	3.5	19.5	2.5	* 4.5	....	....	....	....
509	18.5	42.5	5.5	41.0	4	24	2	* 5.0	....	....	....	....
F-1	12.5	25.5	7	* 7	2	4.5	3	3.5	....	....	....	....
F-2	14.5	21	13	25	3	* 5	2	3.5	....	....	....	....
Basic Open Hearth Steels												
9551	22	49	....	....	20	36.5	3.5	* 8	....	....	....	....
9567	22	50	....	....	19.5	34.5	....	....	2	* 5	....	....
O-1	12	40.5	16	25	4.5	* 7	2.5	5	....	....	....	....
O-2	....	....	14	31	3.5	* 6.5	2.5	5.5	....	....	....	....
Acid Electric Steels												
715	....	....	4	30	2	33	2	* 4.5	....	....	....	....
724	16	46.5	18	* 8	3.5	5.5	2	4.5	....	....	....	....
1009	....	....	5	21.5	2.5	* 5.5	1	4	1	3	....	....
1025	13.5	37.5	17.5	* 6	14	3	1	3	....	....	....	....
Basic Electric Steel												
E-1	....	....	7	13	5	12	....	....	2.5	* 7	....	3.5
Triplex Steels												
H-30	....	....	11.5	27.5	9.5	24	....	....	11	21	....	19
H-34	....	....	11	27	9	19.5	....	....	8.5	21.5	....	16
H-38	....	....	10	22	10	19	....	....	5.0	16	5.5	* 5.5
H-42	....	....	7	18	12	16	....	....	4.0	11.5	2.0	* 10
Converter Steels												
1	....	....	14	28.5	16	29.5	....	....	15	19.5	12	20.5
2	....	....	23	44	20.5	39.5	....	....	16	29	14.5	* 4.5
3	....	....	20.5	39.5	18.5	25	....	....	17.5	27	14.5	29
4	....	....	21	37.5	17.5	35.5	....	....	15.5	* 5	9	3.5
A	....	....	14.5	29.5	9.5	20.5	....	....	8.5	13	3.5	* 3
B	....	....	9	14	7	17.5	4.5	12	5.5	16	2	* 7
C	....	....	7.5	14	....	....	2.5	12.5	2	* 6.5	....	....
D	....	....	11.5	33	11.5	22	....	....	9.5	22	5	16.5

Strain Age-Hardening Effects:

The ability of a material to retain its impact strength after strain age hardening is often considered of importance in evaluating it for some applications. Converter steels have been considered to be especially susceptible to strain age-hardening effects because of a supposedly high nitrogen or oxygen content. In view of this, a number of comparative tests were made on the cast steels.

Specimens of the normalized and drawn coupons were machined to oversize dimensions for the standard impact bar. These were then compressed in the center of the specimen for about $\frac{1}{4}$ in. on either side of the notch location until they were cold worked 10 per cent. The specimens, after the cold working treatment, were next finish machined to size and the keyhole notch put in the center of the worked portion. Four specimens were machined from each steel, two of which were tested as cold worked. The duplicate pair of specimens were aged at 475 F. for 30 min. and then tested at room temperature. Average results obtained in the impact tests are shown in Table V. Rockwell "B" hardness data are also given in the table, and it will be noted that the major increase in hardness of the steels occurs after cold working. The aged specimens

TABLE V.—EFFECT OF STRAIN-AGING ON NOTCHED-BAR IMPACT PROPERTIES.

Steel Number	Normalized and Drawn		Cold Worked 10 per cent		Cold Worked 10 per cent and Aged[a]		Reduction in Impact Strength, per cent		Deoxidation Practice Special Deoxidizers
	Charpy, ft-lb.	Rockwell "B" Hardness	Charpy, ft-lb.	Rockwell "B" Hardness	Charpy ft-lb.	Rockwell "B" Hardness	After Cold Working	After Cold Working and Aging	
ACID OPEN HEARTH STEELS									
493	25	79	17	88	12	88	32	52	Fe Ti and Ca Si
509	24	81	16.5	90	5	90	31	79	Fe Ti and Ca Si
F-1	18	85	10	91	5	94	44	72	None
F-2	18	86	10	94	4	95	44	78	None
BASIC OPEN HEARTH STEELS									
9551	27	80	20.5	88	16.5	89	27	39	None
9567	26	80	21	89	16	91	19	39	None
O-1	22.5	83	16	92	10	94	29	56	None
O-2	24	83	16.5	92	8	94	31	67	None
ACID ELECTRIC STEELS									
715	20	84	18	91	9	92	10	55	Fe Ti and Ca Si Mn
724	23	83	15	91	9	91	35	70	Fe Ti and Ca Si Mn
1009	20	85	5	92	5	95	75	75	Fe Ti and Ca Si Mn
1025	21.5	81	12	92	4	94	44	81	Fe Ti and Ca Si Mn
BASIC ELECTRIC STEELS									
E-1	12	91	7	96	4	99	42	75	Al 0.033%
CONVERTER-ACID ELECTRIC STEELS (TRIPLEX)									
H-30	19	84	15	89	12	92	21	37	Al 0.10%
H-34	18.5	84	13	90	10	94	31	46	Al 0.10%
H-38	15	82	10	88	8	92	33	47	Al 0.10%
H-42	13	86	10	92	6.5	95	23	50	Al 0.10%
CONVERTER STEELS									
1	21	80	15.5	90	15	90	26	29	Al 0.10%
2	28	78	24	87	17	87	14	39	Al 0.10%
3	26	79	20	88	18	89	23	31	Al 0.10%
4	25.5	76	19	86	17	87	25	30	Al 0.10%
A	23	81	11	90	4.5	92	52	80	Al 0.05%
B	13	87	6.5	94	6	94	50	54	Al 0.05%
C	13	84	10	92	8.5	93	23	35	Al 0.05%
D	23.5	82	13	90	11.5	92	45	51	Al 0.110%

[a] Aged 30 min. at 475 F.

show only minor differences in comparison with the cold-worked specimens as a result of the aging treatment.

Impact strength values, on the other hand, show marked reduction, the major portion of which occurs in some of the steels after cold working and in others after aging. This is best illustrated in the percentage of reduction figures shown in the table. It will be noted, in comparing these data with the deoxidation practice used, that some relationship is evident. Those steels having no strong deoxidation treatment, as a rule, are much more susceptible to aging after cold working than are the steels fully deoxidized. It will be observed in the data that the former steels show substantially larger decreases in impact strength after aging than do the latter steels. The steels receiving the strong deoxidation have the major reduction in impact strength occurring after cold working, as will be observed in the tests on the converter group of steels. The two basic open-hearth steels, 9551 and 9567, are apparent exceptions to this rule and show good resistance to strain aging.

The effect of deoxidation practices on the aging tendencies of these steels has a corollary in its effect on aging in deep-drawing steels. In the rimming steel grade of deep-drawing steels, no aluminum is used and the steel is strongly susceptible to aging after the temper rolling following the annealing operation. When this grade of steel has been killed with aluminum, however, it loses its aging characteristics to a large extent and is known as a nonaging killed steel.

Fatigue Properties:

The fatigue or endurance strengths of the grade B steels has assumed an importance greater than normal during the present emergency. Very few data are available on cast steels which could be used in comparing the various steels produced by the different melting units. A few tests were, therefore, made on representative steels from each group received. In order to obtain a fairly complete analysis of the fatigue properties of the steels, two types of specimens were run in the tests. These were standard smooth specimens and Kommers square-notched specimens. Two of the steels, the acid electric steel 724 and

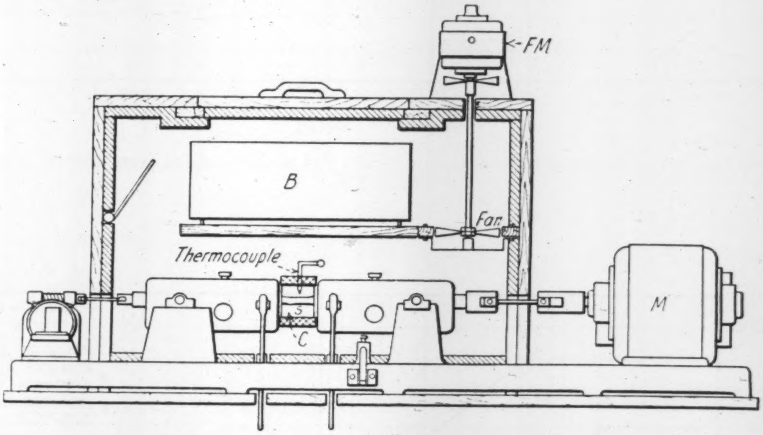


FIG. 6.—Diagrammatic Sketch of Low-Temperature Fatigue Equipment.
B—Dry ice basket.
C—Enclosed chamber. Temperature controlled by thermocouple.

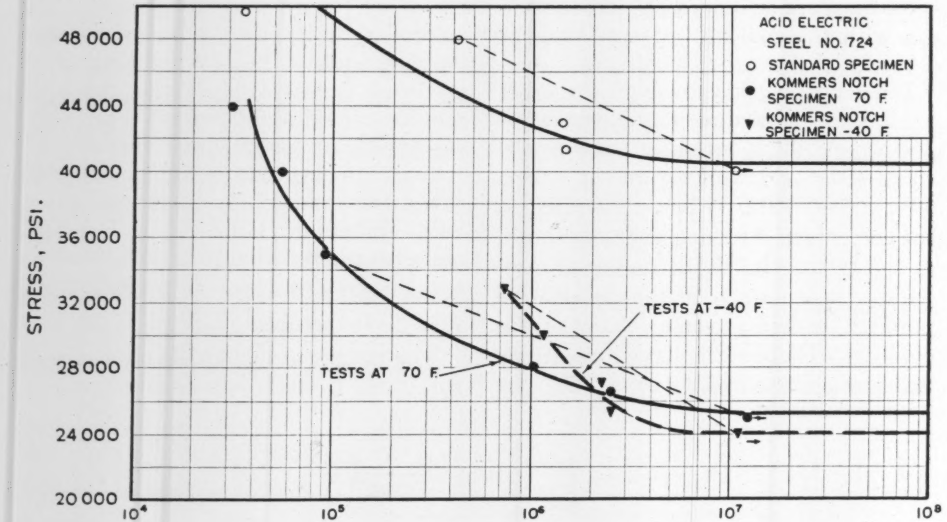


FIG. 7.—Fatigue Curves for Acid Electric Cast Steel 724 as Normalized from 1650 F. and Drawn at 750 F.

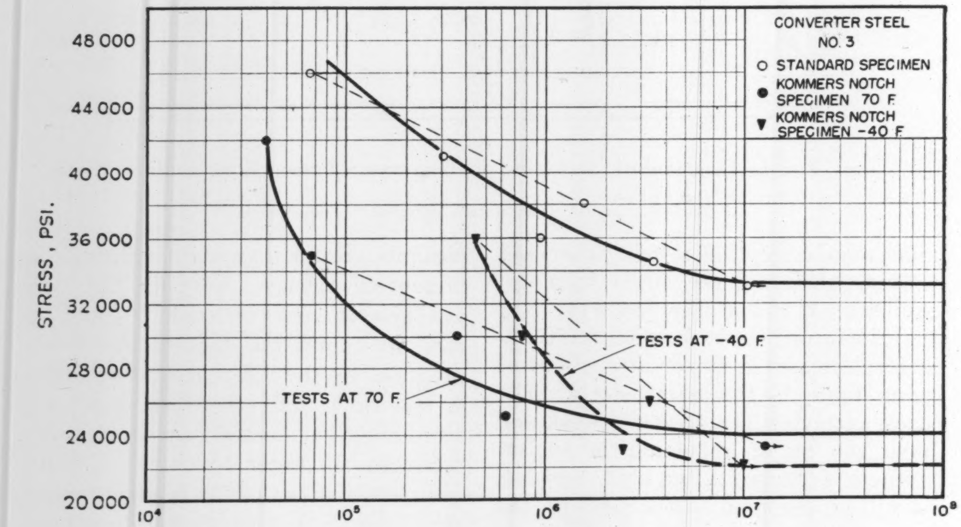


FIG. 8.—Fatigue Curves for Converter Cast Steel 3 as Normalized from 1650 F. and Drawn at 750 F.

converter steel 3, were also tested at −40 F., using the notched specimens only. A cross-section of the low-temperature box used in the tests is illustrated in Fig. 6. Similar low-temperature tests were run previously at Battelle and the operation of the equipment is described in detail in a paper by Russell (**10**).

The fatigue data obtained are shown complete in Table VI, and fatigue curves for two of the steels are plotted in Figs. 7 and 8. Data for the standard specimens show a distinctly higher endurance ratio for the two acid steels than for the basic and converter steels. Inasmuch as only one heat from each group was run, however, no generalization can be made.

TABLE VI.—FATIGUE PROPERTIES OF GRADE "B" CAST STEELS.

Steel	Endurance Limit of Steels, psi.[a]			Endurance Ratio, Endurance Limit/Tensile Strength		
	Unnotched at Room Temperature	Notched at Room Temperature	Notched at −40F.	Unnotched at Room Temperature	Notched at Room Temperature	Notched at −40F.
Acid open hearth steel No. 493	38 000	22 000	....	51	29.5	....
Basic open hearth steel No. 9551	35 000	22 000	....	46	29.0	....
Acid electric steel No. 724	40 000	25 000	24 000	50	31.0	30.0
Basic electric steel No. E-1	40 000	27 000	....	43	29.0	....
Triplex steel No. H-30	34 500	26 000	....	40	30.0	....
Converter steel No. 3	33 000	24 000	22 000	43	31.0	28.5

[a] Kommers square notch specimens used at room temperature and at −40 F.

The notch endurance tests at room temperature, on the other hand, have a striking uniformity which shows no preference for any group. Likewise, the two steels tested as notched bars at −40 F. give very similar results. Since Steel 724 had only 2 ft-lb. Charpy impact resistance at −40 F., it is evident, as might have been expected, that fatigue properties do not reflect the type of brittleness brought out by single-blow notched-bar impact tests but, instead, fall in line with the static tensile properties. All of the endurance limits are within normal expectations and do not appear to be influenced by deoxidation practices in the same manner as some other properties tested.

SUMMARY OF RESULTS

Commercial cast steels made by six processes of melting were compared as to composition and mechanical properties. The compositions of all were normal.

The basic open-hearth steels had lower phosphorus contents. If the identification were to be removed from the others, it would be difficult to decide by what process they had been made.

The converter steels, as a group, were deoxidized with aluminum, whereas only two of the other steels were aluminum killed.

The microstructures were governed by the deoxidation practice, both as to inclusion type and grain size. Aluminum was the only deoxidizer that caused a definite change in type of inclusion. Titanium produced some grain refinement but not so much as aluminum.

The room temperature mechanical properties were entirely orthodox for grade B cast steel, and varied with the composition and type of inclusions, without regard to the method of manufacture.

The converter steels, as a group, had the best low-temperature notched-bar impact properties, but this is held to be a result of deoxidation with aluminum rather than melting practice.

The aluminum-killed steels also showed less strain age hardening, which gave a fallacious advantage for the converter steels.

A limited number of standard fatigue tests showed a superiority in endurance ratio for the acid open-hearth and acid electric steels, but notched fatigue tests showed a notable similarity for all steels.

CONCLUSIONS

No evidence was found to favor one process over another, except that the basic open hearth gave better phosphorus control. Mechanical or engineering properties of all the steels were governed by composition and by deoxidation, rather than by melting process. For example, acid electric cast steel not having had an adequate aluminum addition shows low-temperature impact shortcomings, just as converter steel would without an adequate aluminum addition. When low-temperature shock resistance is needed, it would be in order to impose a low-temperature notched-bar impact acceptance test. If this be done and steel be passed or rejected on performance rather than on the melting process used, some steel now being accepted but not of good low-temperature toughness would be excluded and much good converter steel would be made available.

With proper deoxidation, all the processes can make steel of the quality demanded for war purposes.

REFERENCES

(1) W. C. Hamilton, "General Survey of the Steel Castings Industry," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part II, p. 45 (1932).

(2) J. A. Osborn, "The Steel Foundry Industry in 1935," *Foundry Trade Journal*, Vol. 54, January 9, 1936, pp. 26–27.

(3) C. E. Sims and F. B. Dahle, "Effect of Aluminum on the Properties of Medium Carbon Cast Steel," *Transactions*, Am. Foundrymen's Assn., Vol. XLVII, pp. 65–132, 1938.

(4) C. E. Sims and G. A. Lillieqvist, "Inclusions—Their Effect, Solubility and Control in Cast Steel," *Foundry Trade Journal*, Vol. 47, August 11, 1932, pp. 77–82; *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 100, p. 154 (1932).

(5) H. W. Gillett, "Impact Resistance and Tensile Properties of Metals at Subatmospheric Temperatures," Project 13, Joint A.S.M.E.–A.S.T.M. Research Committee on Effect of Temperature on The Properties of Metals, p. 33, Am. Soc. Testing Mats. (1941). (Issued as separate publication.)

(6) C. H. Herty, Jr., and D. L. McBride, "Effect of Deoxidation on The Impact Strength of Carbon Steels at Low Temperatures," *Cooperative Bulletin 67*, Carnegie Institute of Technology and Mining and Metallurgical Advisory Boards, 52 pp., (1934); Abstracted in *Metals and Alloys*, Vol. 6, March, 1935, pp. 71–77.

(7) A. B. Kinzel, Walter Crafts and John J. Egan, "Fine Grained Structural Steels for Low-Temperature Pressure Vessel Service," *Metals Technology*, Vol. 4, April, 1937, 24 pp.

(8) S. L. Hoyt, "Notched-Bar Testing and Impact Testing," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part II, p. 141 (1938).

(9) D. J. McAdam, Jr., and R. W. Clyne, "The Theory of Impact Testing: Influence of Temperature, Velocity of Deformation, and Form and Size of Specimen on Work of Deformation," *Proceedings*, Am. Soc. Testing Mats, Vol. 38, Part II, p. 112 (1938).

(10) H. W. Russell, "Effect of Low Temperatures on Metals and Alloys," Symposium on Effect of Temperature on the Properties of Metals, p. 658, Am. Soc. Testing Mats. (1931). (Issued as separate publication.)

DISCUSSION

Mr. A. H. Jameson.[1]—The poor old converter certainly has led an eventful life. For a generation or two she was in good standing in the community—her offspring did good service—and then suddenly she was told she had been living in sin, her children were illegitimate and she was to be relegated to the dog house—and there she lay. And this Society approved that banishment on the basis of no evidence, nor scintilla of evidence, although we asked for it definitely and repeatedly.

Then recently the Government in a Federal Specification came out with the converter omitted, and when I asked why, I was told that Epstein's results had shown that converter steel was unreliable at low temperatures. Epstein's results were based on high-phosphorus, high-carbon, rolled steel made in a bottom-blow vessel, from which it was argued, I suppose, that low-carbon, low-phosphorus, cast steel made in a side-blow vessel was unreliable. I think that's a very choice example of *non sequiter.*

The paper by Sims and Dahle has given us the first actual honest-to-goodness comparison of converter cast steel with cast steel made by other processes, and, as might have been expected they are all good if they are well made. We always contended that any of the processes may make poor steel, that any of them could make good steel, and that the prejudice against the converter was unfounded, and we besought the proponents or the opponents of the converter to bring the evidence. They never did. Now we have it, and I think the authors of the paper are to be congratulated upon the work they have done and the impartial way in which they have presented their results, and that the Society should feel a bit humiliated that for nearly twenty years it has cast aspersions on a process which is good and makes good steel. It made good steel then, and the users have not changed the practice. The practice that made this excellent steel that the authors mentioned was the same practice we were following then, and we have just now gotten to the point where it is recognized as basically sound, and it has been adopted as standard practice by electric furnace foundries.

The company which sponsored the research reported in this paper merits our thanks.

Mr. John Howe Hall.[2]—Mr. Jameson has told you briefly and very truly the actual origin of the slur on converter steel. There is one thing Mr. Jameson said which is not quite in accordance with my view. He said we have not changed our practice in the manufacture of converter steel. Well, that depends upon what period you refer to; previous to the World War we had the sulfur worry, and there was no adequate way of curing it at that time. In a recent article of mine in *The Foundry*[3] a long table of converter steel properties and analyses dated 1909 to 1913 gives sulfur running from 0.056 to 0.101 per cent. Of course, that has been changed by the advent of the method of treating the steel with alkalies which enables the sulfur to be controlled. With that exception the process is the same now as it always was. The phosphorus depends upon the grade of scrap and pig iron used as raw material, and there has always been plenty of converter steel made with phosphorus quite as low as that in acid electric or acid open-hearth steel.

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[1] Manager of Castings Sales, Malleable Iron Fittings Co., Branford, Conn.

[2] Consulting Engineer, Philadelphia, Pa.

[3] John Howe Hall, "A Little Knowledge Is a Dangerous Thing," *The Foundry*, September, 1939, p. 22; October, 1939, p. 31; November, 1939, p. 35.

However, at the time that the American Society for Testing Materials decided to give the converter the "bum's rush" the sulfur elimination method was available, and it was because the sulfur elimination method was available that the converter got put in the dog house. Before that, it would have been a simple matter for Subcommittee XXII on Valves, Fittings, Piping and Flanges for High-Temperature Service, of Committee A-1 on Steel, merely to draw a sulfur requirement that could not be met in the converter, in their Specifications for Steel for Castings for High-Temperature Service, and the converter would have been out; but when a method was developed that permitted us to make sulfur as low as with any other process, then something else had to be done, and as you all know, in 1929, after a very disagreeable 5-yr. row the converter went out. Moreover converter steel went out without one piece of evidence of any name, nature, or description ever being advanced to show that it was in any way inferior in its properties as it was made then, to acid open hearth or electric steel. Electric steel was adopted as the preferred method for manufacturing castings for high-temperature service, with open hearth and "other processes satisfactory to the consumer," also allowed, and you all know what that means.

Acid electric furnace steel at that time was suffering from a disease known as "aluminum embrittlement," and there was far more trouble in 1924 to 1926 in getting acid electric steel with good properties than there was with converter steel, but that fact was not candidly admitted. It was not until the 1930's—largely through the work of Mr. Sims, which he characteristically has not mentioned—that it was learned that the cure for aluminum embrittlement in acid electric steel was more aluminum. Mr. Sims and his associates about that time also outlined the proper method of making good acid electric steel, and as I have pointed out elsewhere, the metallurgical features of that method were practically the same as what happens automatically in the converter. Therefore, it is not surprising, as Mr. Jameson says, that when you get some real facts you find that all well-made steels are on a par.

The A.S.T.M. was largely responsible for this slur on the converter which has taken so long to eliminate, and which I do not hesitate to say has been just too bad this year, with the war emergency facing us, and the demand for steel castings far outrunning the supply.

I maintain that steel process clauses in A.S.T.M. specifications are highly undesirable.

MR. F. A. MELMOTH[4] (*by letter*).—In view of the continuous and growing discussion which has taken place on this particular subject during the past year, this contribution to our knowledge of converter cast steel is particularly apt.

I have for a considerable period numbered myself among that group of steel foundry technicians who have vigorously contended that the converter has a definite place in the steel foundry, and that it can demonstrate, on the quality of the product produced, its right to that place. Its omission from certain specifications has reduced the benefit in quick increase of output which could have resulted from the acceptance of its product under adequate inspection and testing safeguards.

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[4] Vice-President, The Detroit Steel Casting Co., Detroit, Mich.

The authors are to be congratulated on the orderly and logical way in which they have carried out their comparative series of tests, and for the definite conclusions they have drawn based upon demonstrated facts, which is a refreshing substitute for prejudices, often based largely upon hearsay.

I would ask to be permitted, however, to mention one or two points which, if not thoroughly appreciated, might cause misunderstanding in the minds of people contemplating the various steel-making methods available to steel founders. The sample E-1 basic electric steel shows up extremely poorly in the heat treated condition, which the authors, quite logically in my opinion, utilized as a standard for their comparisons. This treatment, with ordinary carbon steel, naturally produces practically nothing in the way of air hardening, and a drawing temperature is therefore not demanded high enough to be of material assistance in the obtaining of a high combination of physical properties. However, should there exist a sufficient quantity of certain alloys the air-hardening which takes place on normalizing is sufficient to demand an adequate draw, for an entirely different purpose from that which the authors instituted for the purpose of this comparison. This drawing temperature being so low, it was inadequate to produce beneficial results in this case, I presume, because of the existence of small quantities of nickel, chromium, copper, and molybdenum.

While I agree that a definite draw temperature was necessary in order that the comparisons should be logical, yet this has reacted so much to the disadvantage of the basic electric, containing residual alloys, that quite incorrect inferences might be drawn as to its available properties as a steel casting material, a condition which I believe would react unjustifiably where the installation of new steel-making equipment and distinctions of type were being considered. If the comparison is to have a true value it is essential that the analysis of all the different types of steel should be such as would give almost identical response to the normalizing treatment, and I do not feel that this has been the case in this instance.

I realize that a reference is made in the text to the fact that these alloys have affected the physical test, but would suggest that the degree of this effect has been very much higher than the text would lead one to believe, and the point is raised purely with a view to insuring the basic electric process getting fair consideration.

The low-temperature impact properties of the converter steel specimens, particularly when considered in separate ranges of deoxidation, are remarkable and should conclusively dispose of an impression which has been given considerable publicity during the past year, that converter steels as a class are inferior in this connection.

Another striking point which is of real practical interest is the behavior of the converter acid electric steel triplex. There have been many discussions as to the value of this expensive setup as compared to straight converter steel, and I am interested to see confirmation of the comment that I have made on several occasions that the physical properties of steel held in an acid electric furnace after blowing in a converter were liable to several ductility-affecting effects.

The reduction of area of this class,

while acceptable for some purposes, would in three of the four cases be rejected, were the normal steel casting commercial specifications rigidly applied, as most of these latter place a low limit of 36 per cent reduction as necessary. It would appear, therefore, that the fear expressed has some basis in fact.

I should like to offer my personal thanks to the authors for this excellent paper, which must have the effect of clearing up in our minds many of the misapprehensions arising out of an incomplete knowledge.

MR. H. W. MAACK[5] (*presented in written form*).—As users for twenty-five years of the basic electric process we do not consider heat E-1 to be representative of the steel made by this process, nor the scant data given to permit a correct evaluation of the quality of such cast steel. In these days of scarcity of desirable steel scrap a process which readily controls phosphorus and sulfur contents well within specification limits has practical and economic advantages as well as quality factors.

That the phosphorus content influences shock resistance of steel is probably not to be denied. For the several heats of converter steel tested by the authors, impact values as shown on Fig. 5 vary inversely as phosphorus content. Deoxidation treatment, too, is shown to be a factor although all heats were deoxidized with aluminum. Phosphorus content of about 0.02 per cent is regularly realized in basic process electric steel.

Time did not permit the preparation and testing of specimens of basic electric steel in the same manner as done by the authors. However, some results of Charpy tests at low temperatures are available. These, the average for three normalized-and-drawn heats of about 0.30 per cent carbon cast steel, are as follows:

At 75 F.	21.2 ft-lb.
At 0 F.	17.6 ft-lb.
At −30 F.	12.2 ft-lb.
At −50 F.	9.8 ft-lb.

Note particularly the superior value at −50 F. compared with open-hearth steels in Fig. 5, probably due to the use of 1 lb. of aluminum per ton in the basic electric steel.

MR. A. W. DEMMLER[6] (*presented in written form*).—About the time that the data for this paper were being assembled, I learned that Mr. Sims was quite interested in obtaining low-temperature impact results on Bessemer practice S.A.E. 1040 steel. We had some room temperature information on a 20-ton heat in which one 6700-lb. ingot was treated in the mold with a 4-lb. per ton addition of Grainal No. 1. This heat analyzed:

Carbon, per cent	0.44
Manganese, per cent	0.91
Silicon, per cent	0.20
Sulfur, per cent	0.039
Phosphorus, per cent	0.074

Jominy hardenability of the base heat was 0.16 in. to 50 Rockwell C, while the corresponding figure for the Grainal treated material was 0.29 in. Our original results on material quenched from 1525 F. in oil and tempered at 450 F. were:

Steel	Yield Point, psi.	Tensile Strength, psi.	Elongation, per cent	Reduction of Area, per cent	Brinell Hardness	Izod Impact, ft-lb.
1650 F. (½ hr.)N. + 1525 F. (⅓ hr.)O.Q. + 450 F. (1 hr.) Air						
Plain	150 250	196 500	1.0	3.1	325	2.5
Grainal	226 600	277 100	8.5	30.4	522	10.7

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[5] Chief Chemist and Metallurgist, Crane Co., Chicago, Ill.

[6] Metallurgical Engineer, Vanadium Corporation of America, New York, N. Y.

The impact figures in the above are for the standard izod specimen.

For low-temperature work, we have consistently held to the keyhole notch Charpy specimen. In order to fit into the heat treatment procedure of the paper just presented, we normalized 1-in. rounds from 1650 F. and tempered them at 750 F., subsequently machining the Charpy specimens centrally from the bars. The following low temperature results were obtained:

Steel	Charpy, ft-lb.			Vicker (30-kg. load)
	70 F.	0 F.	−50 F.	
Plain	16.7	8.7	5.5	199
Grainal	20.2	16.0	12.7	192

MESSRS. C. E. SIMS[7] AND F. B. DAHLE[7] (*authors' closure by letter*).—The discussions of Messrs. Jameson and Hall indicate that converter steel was probably condemned unjustly. However that may be, the present paper shows that the product of the converter not only is steel but acts like steel in its response to changes in composition and variations in heat treatment. It should be judged, therefore, by the same standards as steels made in an apparatus with another name, but which utilizes the same fundamental principles.

Mr. Jameson's statement that the converter practice has not changed might be somewhat misleading, for certainly the technique has improved. One of the reasons it took so long to train the old-time blower was that he had to learn to make rapid adjustments for the many variations in temperature and composition of the charge to the converter. Having a known or a constant charge in the converter is half the battle in turning out a consistent product. This is possible today through modern methods of chemical control and pyrometry.

The authors are in complete accord with the contention of Messrs. Melmoth and Maack that the basic electric heat E-1 is not a good representative of its class. It is objectionable on the grounds that both the sulfur and phosphorus contents are too high for the best basic practice and its alloy content puts it outside the carbon steel classification. It might well have been omitted from the data, but it must be apparent that the shortcomings of this steel result from composition rather than method of manufacture. Mr. Maack's data on basic electric steel are a welcome addition.

As a matter of fact the whole study could have been rounded out better had there been an opportunity to secure a second lot of steels specified to fill the gaps in the data from the first lot. Particularly some open-hearth and electric steels deoxidized with aluminum and some converter steels with no addition of aluminum would have been valuable. All of the coupons used, however, were furnished gratis by the producers at some cost and trouble to themselves. This was greatly appreciated and besides the lack of time, there was reluctance to impose on them further.

The data submitted by Mr. Demmler give gratifying confirmation of the present findings. They show that bottom-blow Bessemer steel is also free from notch brittleness at subnormal temperatures when properly deoxidized.

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[7] Supervising Metallurgist, and Assistant Supervisor, respectively, Battelle Memorial Institute, Columbus, Ohio.

EVALUATION OF FATIGUE DAMAGE OF STEEL BY SUPPLEMENTARY TENSION-IMPACT TESTS

By J. A. Kies[1] and W. L. Holshouser[1]

Synopsis

A more complete knowledge of the nature of changes occurring in metals during repeated stress in the unsafe range seems to be required before a nondestructive field test can be devised which will foretell the coming of fatigue cracks. Many attempts have been made to provide such information by various means of which impact testing is one. Thus far the use of impact testing for this purpose has been done under conditions far from optimum.

One purpose of the present paper is to examine the merits of tension-impact testing for investigating some of the changes occurring during repeated stressing in the unsafe range before cracks start. Another purpose is to evaluate the deleterious effects of fatigue cracks of various sizes and at various temperatures.

Smooth (unnotched) Haigh fatigue specimens were subjected to repeated axial loadings in the unsafe range. Some of the specimens were removed from the fatigue machine before cracks could be detected and others after cracks of various sizes had developed. Tension-impact tests were then made at room temperature and at −33 C.

Elongations during tension impact and energies absorbed during tension-impact fracture were related to permanent sets acquired during repeated stressing for those specimens in which no cracks were found. For those specimens containing visible cracks the relations between crack dimensions and the impact elongations and impact energies were studied.

It is concluded that no appreciable losses in elongation during tension impact and no appreciable losses in tension-impact energy were found in uncracked specimens except in cases where considerable permanent set had been acquired during repeated stress. It was also concluded that the smallest detectable fatigue cracks caused important losses in impact resistance. It was further concluded that fatigue cracks reached an easily detectable, critical size before a divergence between the tension-impact behavior at room temperature and at −33 C. was encountered.

It is widely recognized that practically all service failures in load-bearing members of high-speed machinery are fatigue failures. Aircraft are particularly vulnerable in the sense that failure without warning of a working part in flight may result in the total loss of plane and occupants. In spite of improvements in materials and design and other essentials such as machine work, it is necessary to exercise constant vigilance in detecting fatigue-damaged parts in order to secure dependability of modern aircraft.

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[1] Associate Metallurgist, and Junior Metallurgist, respectively, National Bureau of Standards, Washington, D. C.

The problem of detection of fatigue damage in service members at present consists of locating cracks. It is the internal changes leading to the opening of a crack and the effects of the cracks themselves which constitute the scope of this discussion. It is not intended to include here a discussion of surface conditions resulting in weakness such as corrosion and frettage. In addition to such external and visible symptoms of impending trouble, it is taken for granted by many that some subtle, and as yet unexplained internal change in the metal precedes the opening of a fatigue crack. In support of this assumption one has only to review the history of any of a large number of fatigue failures in which a load-bearing part functioned perfectly and without visible deterioration or deformation for a long time prior to the beginning of a fatigue crack. Once a fatigue crack has attained an easily detectable size the part may be expected to fracture completely after a short time of additional use, usually amounting to only a small percentage of its total life. Such a situation makes it difficult to apprehend the danger in time, and, for this reason, it is extremely desirable to find some way of foretelling the appearance of fatigue cracks, the earlier the better. In order to be of real use an inspection method for this purpose must be nondestructive.

Existing theories of fatigue offer little help in pointing out an approach to the problem. X-ray diffraction studies have not proved useful for detecting pre-crack fatigue damage in engineering materials under actual service conditions. Measurements of hardness and damping and studies of slip lines and deformation bands although valuable in supplementing our knowledge of the effects of repeated stressing are apparently not being used as routine procedures in detecting fatigue damage even under laboratory conditions.

It seems evident, therefore, that before a nondestructive test for fatigue damage can be devised, further information on progressive changes occurring during repeated stress within the metal must be gathered by every available means. To this end, a long-range program is in progress at the National Bureau of Standards, and the use of impact tests as a possible means to detect damage in the early stages of its progress is an essential part of the program. In choosing impact testing, it was held in mind that results of such tests might be used to indicate such hypothetical progressive changes as the growth of systems of submicroscopic flaws of unknown nature and the building up of strains to the point of near rupture in localized volume elements.

Fatigue cracks are probably the sharpest kind of notch that it is possible to prepare, therefore it seems likely that very small fatigue cracks may cause a sharp loss in the ductility of a structural member under shock loading. The well-known adverse effect of low temperatures on the impact resistance, particularly of ferritic steels, depends for its existence on the presence of a notch or its equivalent. Therefore, it seemed important to include low-temperature impact tests in the investigation of fatigue damage in the hope of detecting cracks as early as possible.

The results of low-temperature impact tests should also be of interest for the sake of obtaining some idea of the order of magnitude of loss to be expected in resistance to suddenly applied loads, especially since aircraft are frequently required to operate where free air temperatures as low as -55 C. and at times as low as -65 C. are encountered. From the point of view of interest in the impact resistance itself, it is important to study the effects of fatigue cracks of various sizes beyond the smallest detectable sizes because, although machine-made notches have been widely used in impact specimens, there seems to be little information available on the effects of fatigue cracks on impact resistance.

A few attempts to use impact tests to indicate progressive fatigue damage have been made. Oshiba (**1**, **2**)[2] experimented with annealed carbon steels and reported losses in impact resistance following repeated stresses both below and above the fatigue limit. Kies and Quick (**3**) found no loss in notched-bar impact energy resulting from repeated stresses below or above the fatigue limit for aluminum alloy 25S-T. Their impact specimens were cut from axial-loading fatigue specimens in which the stress amplitude was fairly uniform throughout the portion of the fatigue specimen from which the impact specimen was cut. Portevin (**4**) also reported negative results of experiments on a crankshaft steel previously stressed as a rotating cantilever beam. In specimens of this kind the outermost layers are subjected to the highest stresses, whereas the stress at the center is zero. It is difficult to see how notched bars of rectangular section could be expected to yield much information when machined from the inner portions of such fatigue specimens.

The uncertainties in stress distribution in transverse impact tests of notched specimens of rectangular section probably far outweigh, and mask, any differences caused by previous fatigue stressing.

Testing of smooth Haigh fatigue specimens in tension impact after repeated stressing and without further machining holds some advantages as follows:

1. Better uniformity and control over the geometry of the specimen and its surface finish is possible during machining of unnotched specimens.

2. Larger volumes of metal are subjected to the maximum stresses than in rotating-beam specimens.

3. Surface layers of greater area in which the maximum damage is to be expected are in a position to influence the impact result than in any impact test specimen in which a notch has been machined or the surface layers removed subsequent to the repeated stressing.

4. The mean stress as well as the stress amplitude can be adjusted on the Haigh machine. It is therefore possible to simulate commonly found service conditions in which alternating loads are superimposed on steady loads.

5. Many test results have shown that the scatter in results of tension-impact tests on smooth unstressed specimens is slight in comparison with transverse tests on notched specimens, at least for normalized S.A.E. X4130 steel. This should make detection of damage in individual cases more certain.

6. It is possible to measure the elongation in a tension-impact fracture, which may furnish additional information not apparent from a comparison of impact energy values.

7. No difference in tension-impact energy is to be expected at room temperature and at -33 C. in the case of undamaged smooth Haigh specimens of normalized S.A.E. X4130 steel. This simplifies the interpretation of impact results for damaged specimens.

In a search for methods of detecting fatigue damage, it is easy to forget the important and obvious fact that a piece of material damaged in one sense may not be damaged at all in other respects. Specifically, numerous "damage line" data have been published following the method of French (5) in which it was clearly demonstrated that metals may be so damaged by a given stress above the fatigue limit that their remaining endurance at the same stress is reduced considerably and yet the fatigue limit is not lowered. In other words the material is apparently damaged only in the sense that it cannot withstand overstress as well as before. Likewise, supplementary tests, for example, impact tests, showing no loss in impact resistance, do not necessarily mean that no fatigue damage exists.

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[2] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 564.

MATERIAL AND SPECIMENS

The material used in this investigation was S.A.E. X4130 (chromium-molybdenum) steel in the form of $\frac{1}{2}$-in. square hot-rolled bars. Before machining into specimens the bars were sawed into lengths of 12 to 18 in. and normalized by holding for 1 hr. at 1650 F. and cooling in still air indoors. All tests were made on specimens having an over-all length of $4\frac{1}{2}$ in. and a minimum diameter of 0.200 in.; the sweep radius was $9\frac{7}{8}$ in., and the ends were threaded (size $\frac{7}{16}$ in., 20 threads per inch). A drawing of a Haigh specimen of the design used and a description of the Haigh fatigue machine have been published by France (6). The test portions of the specimens were finished by longitudinal strokes with Behr-Manning 1G emery paper followed by similar treatment with aloxite 400A paper made by the Carborundum Co. A few of the specimens were given additional polish by the use of Behr-Manning 2/0, 3/0 and 4/0 emery papers in succession. For the purpose of the present paper, there was no distinguishable difference traceable to the two polishes. A discussion of possible effects of polish is not within the scope of this paper; therefore no further reference will be made to the subject.

PROCEDURE

Prior to impact testing, the specimens were separated into groups and were subjected to repeated stressing in the Haigh axial loading machine at room temperature and at unsafe ranges as follows: group (*a*), 15,200 psi. compression to 77,300 psi. tension; group (*b*), 10,500 psi. tension to 89,500 psi. tension; group (*c*), 27,000 psi. tension to 127,000 psi. tension. All three ranges were sufficient to cause fatigue failure within 300,000 cycles. The specimens were removed from the machine after various numbers of cycles, some before cracks could be detected, and some after cracks of various sizes had formed. Some of the specimens fractured completely in the fatigue machine. In addition a group (*d*) of Haigh specimens were subjected to various numbers of cycles of repeated stress in the range 50,000 psi. compression to 50,000 psi. tension which was sufficient to cause failure within 1,000,000 cycles.

The tension-impact tests were made with a 224 ft-lb. Charpy machine at room temperature and at -33 ± 2 C. The threaded ends of the Haigh specimens were screwed into a special hole in the back of the pendulum. A special set of stops for the impact machine and a cross piece were made for this investigation. The geometry of the arrangement was such as to minimize bending of the specimens during ductile impact fractures.

To obtain the low temperatures, the pendulum with specimen and cross piece attached were immersed in a tank of 50 per cent ethylene glycol, 50 per cent water cooled by fragments of solid CO_2 which were added as needed. Before making the impact test the pendulum and specimen were raised from the bath and the tank was rolled out of the way on a track built for the purpose. The temperature of the bath was measured frequently by a thermocouple. The necessary time interval of immersion and the subsequent rate of temperature rise in the specimens after removal from the bath were determined by separate tests. All low-temperature impact tests were completed from 3 to 5 sec. after removal of the specimen from the bath.

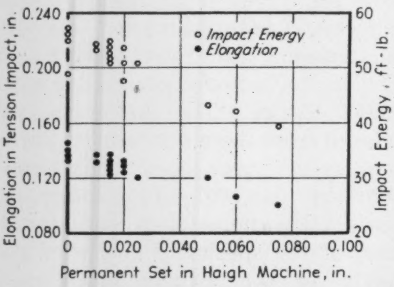


FIG. 1.—The Relation Between Permanent Set During Repeated Stress and the Tension-Impact Properties of Haigh Fatigue Specimens of Normalized S.A.E. X4130 Steel.

The range of cyclic stress was 10,500 psi. to 89,500 psi., all in tension. No fatigue cracks were detected in any of the specimens represented above. Tension-impact fractures were all made at −33 C. The specimens shown as having had zero extension in the fatigue machine were not stressed prior to the impact test. Note that the vertical scale of elongations is compressed by a factor of two in comparison with the horizontal scale of permanent sets in the Haigh machine.

Elongations were determined by fitting together the specimens after impact and measuring the distance between fine punch marks, initially 2 in. apart. All values for elongation as plotted in the various figures are the total extensions of the 2-in. gage portions. The lengths and the maximum depths of penetration of the cracks were measured by means of a Hilger measuring microscope focussed on the fractures.

TEST RESULTS

The Pre-Crack Stage of Fatigue:

When mean tensile stresses and cyclic stress amplitudes high enough to produce appreciable permanent sets were used, there resulted losses in tension-impact energy and elongation even though fatigue cracks were not detected. An example of this was obtained using a repeated stress range of 10,500 psi. to 89,500 psi. Figure 1 shows approximately linear variations of impact energy and elongation with permanent set resulting from repeated stress. The reason for the variations in permanent set represented in Fig. 1 lies in the variations inherent in the specimens themselves as prepared in the normalized condition rather than in different numbers of cycles. The range of repeated stress was the same for all specimens represented in Fig. 1, and although the number of cycles varied considerably, the number exceeding 10,000 in all cases, it is believed that all of the specimens had reached their limits of crackless permanent set before the 10,000 cycles had been completed. Evidence that all of the permanent set occurred within the first few thousand cycles is provided by the fact that the head of the Haigh machine required no further adjustment by the hand wheel after the

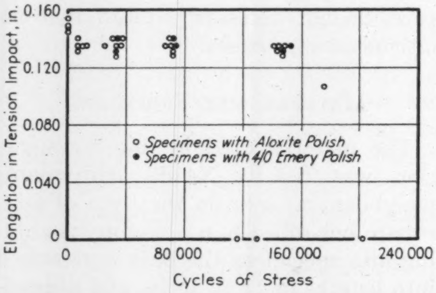
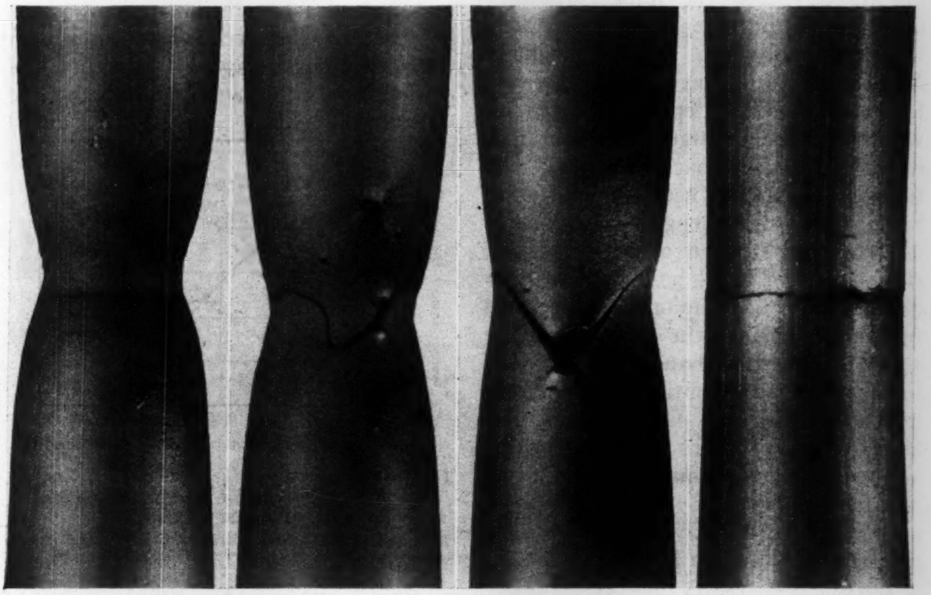


FIG. 2.—The Relation Between Tension-Impact Elongation at Room Temperature and the Number of Cycles of Overstress of Haigh Fatigue Specimens of Normalized S.A.E. X4130 Steel.

Stressed in the range 10,500 psi. to 89,500 psi. all in tension without necking. All specimens represented in this figure as having less than 0.120 in. elongation contained visible fatigue cracks. Some results included in Fig. 1 were arbitrarily omitted from this figure because of necking of the specimens.

first two or three minutes. In further support of this statement, attention is called to Fig. 2 showing the relation between tension-impact elongation at room temperature and the number of cycles of prior stress in the range 10,500 psi. to 89,500 psi., all in tension. It may be noted that after the initial loss in elongation following the first few thousand cycles there was no additional or progressive loss as stressing continues until after cracks were formed. Specimens represented in Fig. 2 as having less than 0.120 in. tension-impact elongation were found to contain fatigue cracks. Tension-impact tests at −33 C. showed the same thing for Haigh specimens stressed the same way although elongations were generally slightly lower. Tests on Haigh specimens overstressed in cycles of equal tension and compression (group *d*) failed to show any drop in impact resistance or elongation before cracking. Furthermore, 31 R. R. Moore rotating-beam fatigue specimens stressed at ±80,000 psi. (fatigue limit = ±60,000 psi.) for various numbers of cycles also failed to show any loss in tension-impact energy or elongation until after fatigue cracks could be detected. A study of the tension-impact energies of uncracked Haigh fatigue specimens showed no clearly distinguishable loss from the initial values regardless of stress range or number of cycles except for those specimens appreciably necked during repeated stress.



Cycles of stress	0	96 000	120 000	146 000
Impact energy, ft-lb	53.5	50.0	33.0	8.0
Elongation, in.	0.150	0.145	0.110	0.020

FIG. 3.—Haigh Specimens of Normalized S.A.E. X4130 Steel Subjected to Repeated Stress from 27,000 psi. to 127,000 psi. All in Tension, then Fractured in Tension Impact at −33 C. (X 5).

The photographs are arranged from left to right in order of increasing degrees of fatigue damage as evidenced by losses n impact energy and elongation. The elongation measurements depended for their accuracy on how truly the positions if the parts as shown represented the case at the time of fracture. All specimens were finished with aloxite paper prior oo repeated stress.
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The Fatigue Crack Stage:

In studying the relation between fatigue crack size and tension-impact properties of Haigh specimens of normalized S.A.E. X4130 steel, only those fatigue cracks contiguous with the impact fracture areas were considered. Most of the impact fractures showed only one detectable fatigue crack entering directly into the impact fracture although many other fatigue cracks were commonly found on the specimen. Measurements of length along the surface and maximum depth of penetration were made for the fatigue cracks entering directly into the impact fractures. The term, crack size, is used hereinafter to mean either crack length or maximum depth of penetration or their product since any one could be shown to be a definite and continuously increasing function of either of the other two within a narrow scatter band. In the few impact fractures contiguous with two fatigue cracks, the dimensions of the larger crack only were used since its size seemed to be the controlling factor in the impact energy. Representative specimens with varying amounts of damage are shown in Fig. 3.

Impact test results on cracked specimens could be related to crack size but not to range of stress previously applied; therefore in plotting results of impact tests on cracked specimens no separation was made according to stress range and results drawn from all three specimen groups are considered together.

The average values for impact energy and elongation were plotted against average values for maximum depth of penetration and for average values of the product of crack length times maximum depth of penetration. The averages were computed for groups of individual test results falling within suitable intervals of magnitude so as to include approximately equal numbers of results in each average. Each test result entered into only one average. Separations or memberships of groups were determined on the basis of crack size only. Class intervals along the abscissae were not equal, but the system used had the advantage of smoothing out the curve in the region of largest crack sizes where the data were relatively sparse. Separate plots using constant class intervals were made but showed no difference in the location of the faired curves and so were not included.

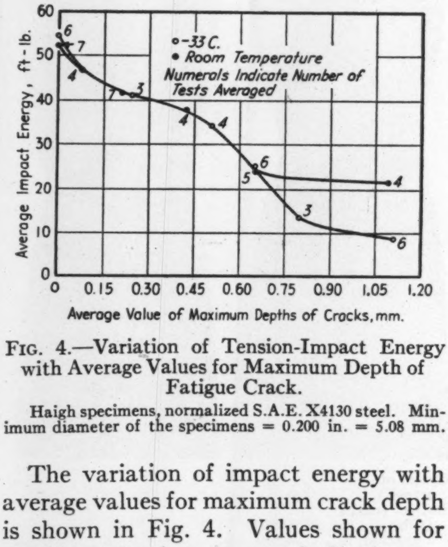

FIG. 4.—Variation of Tension-Impact Energy with Average Values for Maximum Depth of Fatigue Crack.

Haigh specimens, normalized S.A.E. X4130 steel. Minimum diameter of the specimens = 0.200 in. = 5.08 mm.

The variation of impact energy with average values for maximum crack depth is shown in Fig. 4. Values shown for specimens having zero crack depth were taken from tests on specimens not stressed in fatigue. There was no perceptible difference in the results at −33 C. and at room temperatures for the smaller cracks. For cracks more than 0.65 mm. deep, that is, for penetrations of over 13 per cent of the minimum diameter of the specimen, the average impact resistance became less than half the room temperature value. This critical depth at which the low-temperature effect was found corresponded to the second steep part of the curve. The first steep portion corresponded to the advent of the smallest detectable cracks.

The existence of a critical size at which the low-temperature effect begins to show itself is more clearly demonstrated when the factor of crack length is taken into account. Actually the chord defined by the ends of the crack was measured. For the largest crack measured, the arc or crack length, measured along the smooth outer surface of the specimen, exceeded the chord by only 5½ per cent. At the critical crack size the arc exceeded the chord by only 0.7 per cent. These discrepancies are so much smaller than the range of scatter in impact results that the term crack-length will be used instead of chord. Figure 5 shows the variation in impact energy with average value for the product of crack depth times length. For values of this product exceeding 0.70 sq. mm. the loss in impact resistance was more serious at −33 C. than at room temperatures. This critical size corresponded to the steepest portion of the curve with the exception of the initial drop.

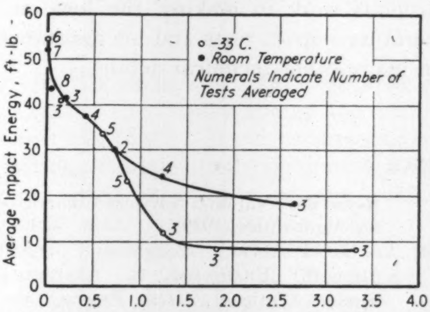

FIG. 5.—Variation of Tension-Impact Energy with the Product of Maximum Depth Times Length of Fatigue Crack Entering Directly into the Impact Fracture.

Haigh specimens, normalized S.A.E. X4130 steel. Minimum diameter of the specimens = 0.200 in. The specimens represented here are the same as those represented in Fig. 4.

Elongations during impact were measured on some of the specimens represented in Figs. 4 and 5. The results obtained were closely similar to the impact energy results. Figure 6 shows an initial steep drop in elongation with the first appearance of cracks and a second steep drop for values of crack depth times length of about 0.70 sq. mm., the same as noted in Fig. 5 for the divergence between room temperature and −33 C. tension-impact energies. This critical crack size corresponds to crack areas of about 3 per cent of the minimum cross-section of the specimen. There is presumably nothing fundamentally significant about the noted magnitude of the critical crack size. From a practical standpoint the important item is that such a thing exists.

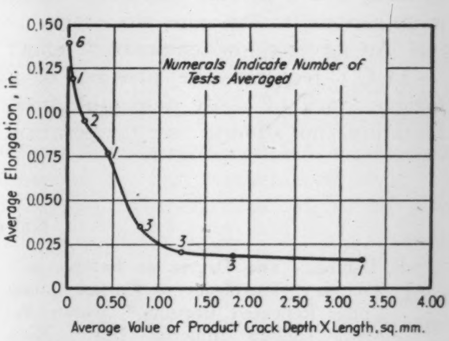

FIG. 6.—Relation Between the Product of Maximum Depth of Fatigue Cracks Times Length and Elongation in Tension Impact at − 33 C.

Note that the steep parts of the curve correspond to those on the impact energy curve of Fig. 5. Haigh fatigue specimens, normalized S.A.E. X4130 steel. The numerals on the curve indicate the number of tests averaged. Minimum diameter of specimens = 0.200 in.

CONCLUSIONS

1. No appreciable loss in tension-impact resistance or elongation during impact resulting from prior repeated overstress in fatigue was noted in Haigh axial-loading specimens in the pre-crack stage except for cases where the mean

tensile stress and stress amplitude were high enough to cause permanent sets of more than 0.01 in. during fatigue stressing. Such conditions are improbable in service and would be easily detected.

2. With the formation of fatigue cracks, however, the situation was changed. Even the smallest detectable fatigue cracks were usually accompanied by substantial losses in tension-impact resistance. Location of a fatigue crack on the tensile side of a specimen in bending would be expected to be much more serious for the same size crack.

3. An adverse low-temperature effect (−33 C.) required the presence of a fatigue crack of easily detectable size; therefore, no adverse low-temperature effect may be expected as a result of repeated stress in the pre-crack stage of fatigue. The critical crack size at which the low-temperature effect may be expected presumably differs with the impact-testing temperature, type of impact-testing machine, specimen size and shape, speed of impact, and possibly other factors.

Acknowledgments:

The authors are greatly indebted to G. R. McConnell, formerly of the National Bureau of Standards, for his arduous work in making the low-temperature impact tests and his assistance in many other technical details.

REFERENCES

(1) F. Oshiba, "The Degree of Fatigue and Recovery Therefrom of Carbon Steels Under Repeated Impacts," *Science Reports*, Tôhoku Imperial Univ., First Series, Sendai, Japan, Vol. 23, pp. 589–611 (1934–1935).

(2) F. Oshiba, "The Degree of Fatigue of Carbon Steels Under Reversed Bendings," *Science Reports*, Tôhoku Imperial Univ., First Series, Sendai, Japan, Vol. 26, pp. 323–340 (1937–1938).

(3) J. A. Kies and G. W. Quick, "Effect of Service Stresses on Impact Resistance, X-ray Diffraction Patterns and Microstructure of 25S Aluminum Alloy," *Technical Report 659*, National Advisory Committee for Aeronautics (1939).

(4) A. M. Portevin, "Toughness (Impact Strength) Unaffected by Alternating Stresses (Fatigue)," *Metal Progress*, Vol. 37, pp. 563–564 (1940).

(5) H. J. French, "Fatigue and the Hardening of Steels," *Transactions*, Am. Soc. Steel Treating, Vol. 21 pp. 899–946 (1933).

(6) R. D. France, "Endurance Testing of Steel: Comparison of Results Obtained with Rotating-Beam *Versus* Axially-Loaded Specimens," *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part II, p. 176 (1931).

DISCUSSION

Mr. W. N. Findley.[1]—The author has observed a measurable difference in impact resistance of specimens containing fatigue cracks when tested at low temperatures as compared to room temperatures. This difference was observed only with specimens containing cracks larger than a certain "critical" size.

In discussing the effect of area of crack on the impact energy the author observes that: "There is presumably nothing fundamentally significant about the noted magnitude of the critical crack size." Perhaps the crack size itself is not fundamental, but it may possibly be related to fundamental properties of the material and test conditions.

The following analysis of the "critical crack size" is largely an hypothesis, but a knowledge of the factors causing brittle fracture is becoming so important that even a conjecture is worth considering.

A sharp notch (such as a fatigue crack) in a tension member tends to set up a state of stress at the root of the notch in which the principal stresses are tension (two of the principal stresses are zero in an unnotched tension member). In such a notched tension member the three principal stresses are all tension but not necessarily of equal magnitude. However as the depth of the notch is increased the three principal stresses in the member at the cross-section of the notch tend to become more nearly equal. Thus the shearing stress becomes smaller and the ratio of the maximum principal stress to the maximum shearing stress, $\frac{\sigma_{\max}}{\tau_{\max}}$, becomes larger ($\frac{\sigma_{\max}}{\tau_{\max}} = 2$ for an unnotched tension member). Now, if the ratio of the technical cohesive strength[2] to the shearing yield strength[2] of the material is smaller at low temperatures than at elevated temperatures; then, as the depth of the notch is increased ($\frac{\sigma_{\max}}{\tau_{\max}}$ increased), a depth of notch may eventually be reached for which the maximum principal stress reaches the technical cohesive strength of the material before the maximum shearing stress reaches the shearing yield strength. Thus the failure will be by brittle fracture rather than by yielding (the failure would be by yielding for smaller notches) with a resulting decrease in energy required to rupture the member.

But if, as postulated above, the ratio of technical cohesive strength to shearing yield strength is higher at room temperature than at low temperature, then tests at room temperature will continue to fail by yielding even when the notch is driven deeper than that which caused the low temperature member to fail by fracture. It might be that at room temperature failure would take place by yielding for all depths of notch, depending on the magnitude of $\frac{\sigma_{\max}}{\tau_{\max}}$ which is obtained and the value of the ratio of technical cohesive strength to shearing yield strength.

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[1] Instructor in Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[2] These values are considered by some investigators to represent the fundamental properties of a material. See paper by D. J. McAdam, Jr., "The Influence of the Combination of Principal Stresses in Fatigue of Metals," p. 576.

Thus the critical crack size may represent the size of crack which produces a ratio $\frac{\sigma_{\max}}{\tau_{\max}}$ such that $\sigma_{\max}$ equals the technical cohesive strength for the lower temperature at the same time that $\tau_{\max}$ equals the shearing yield strength.

Mr. J. M. Lessells.[3]—Regarding the first conclusion made by the author, it is generally agreed that material under cyclic stress and a positive mean stress can show permanent deformation and yet be perfectly safe. The writer believes this conclusion might allow of a misunderstanding that safety under cyclic stress is associated with permanent deformation. This is not necessarily true, as indicated by Gough.[4]

Mr. W. Leighton Collins.[5]—The writer is heartily in agreement with the statement made by the authors "that before a nondestructive test for fatigue damage can be devised, further information on progressive changes occurring during repeated stress within the metal must be gathered by every available means." The writer also believes that since the changes within the metal during fatigue are undoubtedly submicroscopic, a test indicating such damage most probably will have to be highly sensitive to small changes in the material.

The above ideas led the writer to make a few exploratory static torsion tests of virgin steel specimens (no previous stressing) and of specimens which had been stressed at the endurance limit (for completely reversed cycles of torsional stress) for at least ten million cycles of stress without failing. Twisting moment—angle of twist curves for both types of specimens from two different carbon steels were tested.

The initial slopes of the curves and the portions of the curves after marked yielding had taken place were not affected by the repeated stressing. However, in the region of initial marked yielding there were differences such as those tabulated below for a heat-treated 0.59 per cent carbon steel.

Property	Virgin Steel	Previous Fatigue Loading
Proportional Limit, psi.	41 500	22 600
Yield strength, 0.03 per cent offset, psi.	47 200	41 900
Yield strength, 0.15 per cent offset, psi.	51 000	50 400
Yield strength, 0.30 per cent offset, psi.	52 500	52 900

The numerical values for "proportional limit" should not be taken too seriously, but from the above values it is apparent that stressing at the endurance limit caused measurable initial and gradual departure from the straight line portion of the twisting moment—angle of twist curves at lower stresses than were found for the virgin steel specimens.

Since the same testing machine, torsion indicator, testing technique and method of plotting were used for all tests it is felt that the differences noted are significant.

Messrs. J. A. Kies[6] and W. L. Holshouser[6] (*authors' closure, by letter*).—Mr. Findley's discussion is valuable in pointing out the manner in which geometrical considerations may be expected to influence the impact resistance and type of failure of structural parts. In regard to the significance of the noted magnitude of the critical crack size, it must be assumed that every conceivable behavior of a material object is somehow related to those of its physical properties commonly regarded as fundamental. As noted by Mr. Findley, a quantity such as impact resistance or a qualitative response such as brittle fracture depends on those fundamental physical properties and on the geometry of the piece and the conditions of loading. The minimum depth of crack which will produce a brittle fracture in a particular specimen under a particular set of conditions is therefore a derived rather than a fundamental property of the material. The choice of the words "not fundamentally significant" was perhaps not the best, but they were intended to convey the impression that, at least at present, the noted magnitude is not usable in a quantitative estimation on a rational basis of any other property of the material.

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[3] Associate Professor of Mechanical Engineering, Engineering Dept., Massachusetts Institute of Technology, Cambridge, Mass.

[4] H. J. Gough, "The Fatigue of Metals," p. 126, Scott-Greenwood and Sons, London (1924).

[5] Assistant Professor, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[6] Associate Metallurgist, and Junior Metallurgist, respectively, National Bureau of Standards, Washington, D. C.

The results reported by Mr. W. Leighton Collins are interesting because they are different from the findings of Wood and Thorpe[7] who reported that repeated stress suppressed yielding and permanently raised the primitive yield strength of annealed brass. In experiments of this kind there is a complicating factor that may easily be overlooked. That is the effect of starting the cyclic stressing with a low amplitude and gradually working up to that which is reported as *the* amplitude of stress. In Haigh axially loading machines this is almost invariably the case. The resulting effects on the material may be quite different from those obtained by imposing the full amplitude during each cycle starting with the first, particularly if the maximum tensile stress of the cycle exceeds the primitive yield strength.

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[7] W. A. Wood and P. L. Thorpe, "Behavior of the Crystalline Structure of Brass Under Slow and Cyclic Stresses," *Proceedings*, Royal Soc. (London), Vol. 174A, pp. 310–321 (1940).

FATIGUE TESTS AS A MEANS OF EVALUATING CORROSION DAMAGE OF SHEET METALS

BY W. H. MUTCHLER[1] AND J. A. KIES[1]

SYNOPSIS

Flexural fatigue tests, in machines of Krouse design, were used to determine the fatigue limits on stainless steel sheet specimens before and after corrosion by prolonged exposure in marine environments. The tests showed that the rate of corrosion was relatively rapid during the initial stages of exposure, but that thereafter a near arrest in the attack occurred. Panels exposed to the marine atmosphere suffered more damage, for the same period of exposure, than those which were wet periodically by tidewater. The method permitted a reliable evaluation of the damage resulting from corrosion.

It is the purpose of this paper to describe a method utilizing flexural fatigue tests as a means of evaluating the extent of damage resulting from corrosion. The method was applied in an investigation of the corrosion of sheet metals used in aircraft, which is being conducted at the National Bureau of Standards.

Stainless steel panels, approximately 0.018 by 4 by 14 in., were exposed at a marine test site to the weather and to sea water. The panels exposed to the weather were located directly above the water and were within from 6 to 10 ft. of its surface. Those exposed to the salt water were so suspended that they were immersed twice daily during the periods of high tide.

The steels were polished, full-hard, cold-rolled, passivated sheets and were mostly of the 18 per cent chromium, 8 per cent nickel type, with and without the addition of the customary alloying elements such as molybdenum, titanium, and columbium. Inasmuch as this paper deals primarily with the method of test, rather than with the results on the different compositions, the data contained herein relate only to two plain chromium-nickel steels. The results of the tests on the other steels have been published elsewhere.[2,3]

The fatigue tests were used to measure the damage resulting from corrosion in a manner analogous to that in which Basch and Sayre[4] and Blough[5] first utilized tension tests. The fatigue limits were determined on the materials before corrosion and after exposure for different lengths of time, namely, 7½, 12, 24, and 36 months in the tidewater racks and 7½, 24, and 36 months in the weathering racks. All of the exposed specimens showed evidence of corrosion.

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[1] Associate Metallurgist, National Bureau of Standards, Washington, D. C.

[2] Willard Mutchler and W. G. Galvin, "Tidewater and Weather-Exposure Tests on Metals Used in Aircraft," *Technical Note 736*, Nat. Advisory Committee for Aeronautics, November, 1939.

[3] Willard Mutchler and W. G. Galvin, "Tidewater and Weather-Exposure Tests on Metals Used in Aircraft (Part 2)," *Technical Note 842*, Nat. Advisory Committee for Aeronautics, February, 1942.

[4] D. Basch and M. F. Sayre, "Resistance of Various Aluminum Alloys to Salt-Water Corrosion," *Mechanical Engineering*, Vol. 46, p. 199 (1924).

[5] E. Blough, "The Evaluation of Corrosion Tests," *Proceedings*, Am. Soc. Testing Mats., Vol. 25, Part II, p. 156 (1925).

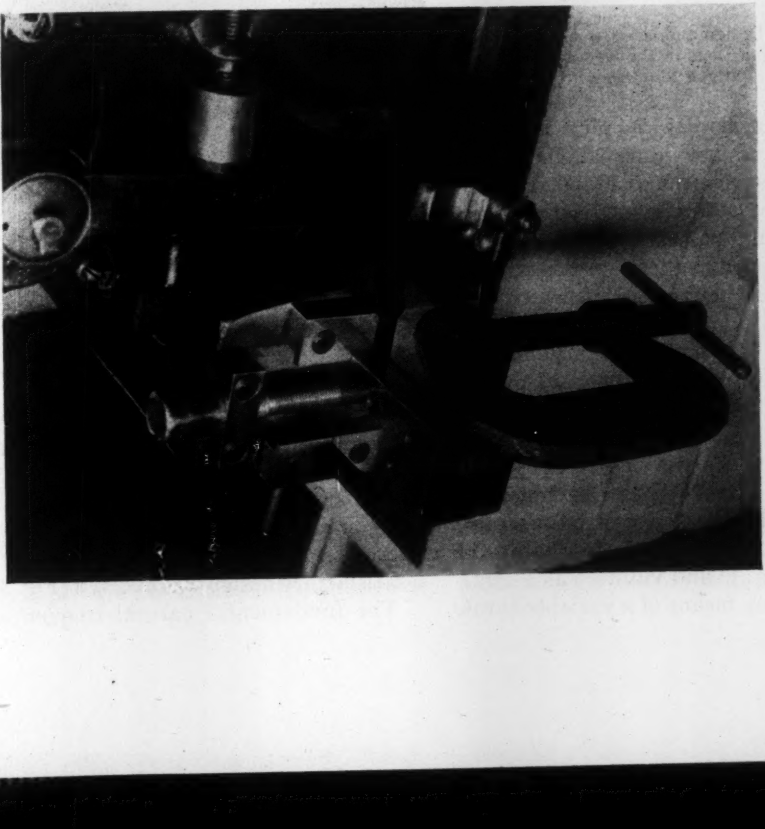

FIG. 1.—A Specimen in the Krouse Flexural Fatigue Testing Machine. Showing the method of attaching it at the fixed and loading ends.

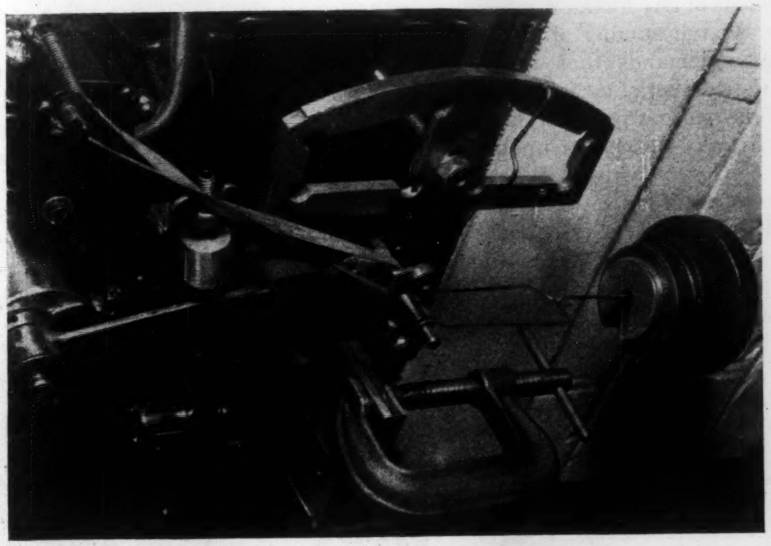

FIG. 2.—A Specimen Loaded with Dead Weights for Determining Its Deflection Preparatory to Calculating the Maximum Stresses. Deflection measurements were made by means of the pointer and the scale on the arc at the right.

In order to establish accurately the fatigue limit of either uncorroded or corroded specimens of any metal, it is necessary to test a large number of samples. Accuracy also demands that tests be made with the number of cycles in excess of 100 million, since values for fatigue limits are partially dependent upon the number of cycles of stress. Where a high degree of accuracy in determining fatigue limits is considered essential, the use of the method which the authors adopted is precluded.

In this work, the fatigue tests were employed, however, solely with the objective of obtaining an approximate measure of the relative rate of damage resulting from corrosion on each of several steels of similar chemical compositions. As is not unusual, both the number of specimens available and the time to be devoted for testing were limited. The values selected for the fatigue limit were therefore based upon a stress, the half range of which was within 800 psi. of the next highest stress which resulted in failure, provided at least two "runs" past 10 or 20 million cycles had been made.

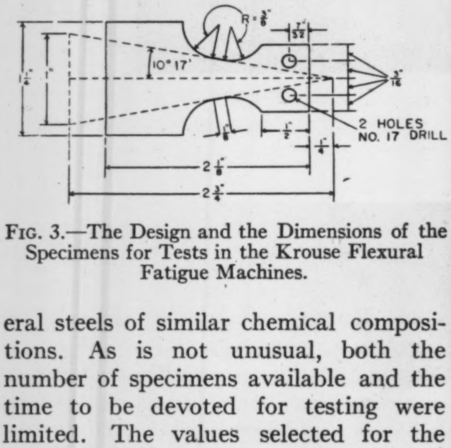

FIG. 3.—The Design and the Dimensions of the Specimens for Tests in the Krouse Flexural Fatigue Machines.

The tests were made in flexural fatigue testing machines developed by G. N. Krouse for sheet. These machines are of the fixed-deflection (constant-strain) type, the specimen being loaded at the free end (Fig. 1) and vibrated as a cantilever beam by means of a variable-throw crank and a connecting rod. The cycle of stress represents a complete reversal from a maximum tensile stress to a compressive stress of equal magnitude.

Each specimen, before testing, was calibrated as its own dynamometer by measuring its deflection when loaded with dead weights (Fig. 2), and by adjusting the variable throw crank to correspond to this deflection. The method of determining the stress on the Krouse machines was improved somewhat by placing the scale used for measuring the deflections on an arc, instead of along a straight line.

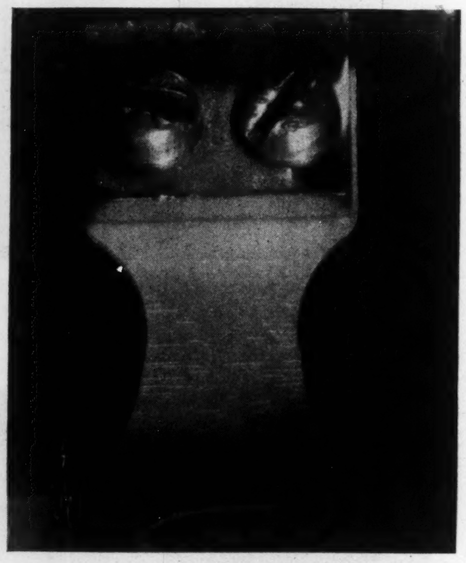

FIG. 4.—Cracks in a Brittle Lacquer Applied to a Flexed Specimen Demonstrated that the Distribution of the Stresses Was Fairly Uniform.

The design for test specimens recommended by the manufacturer was found inadequate for steels having the high tensile properties and the thin gages of those used in the investigation. However, after some preliminary experiments, a satisfactory design was evolved (Fig. 3).

The fundamental natural frequencies

of the steel specimens, as determined with a strobotac, with clamps in place on the Krouse machine, were 1680 to 1695 vibrations per minute for a sample 0.0173 in. thick. These natural frequencies, fortunately, were close to the 1740 vibrations per minute at which the specimens were tested, a circumstance which tended to suppress undesirable bending moments.

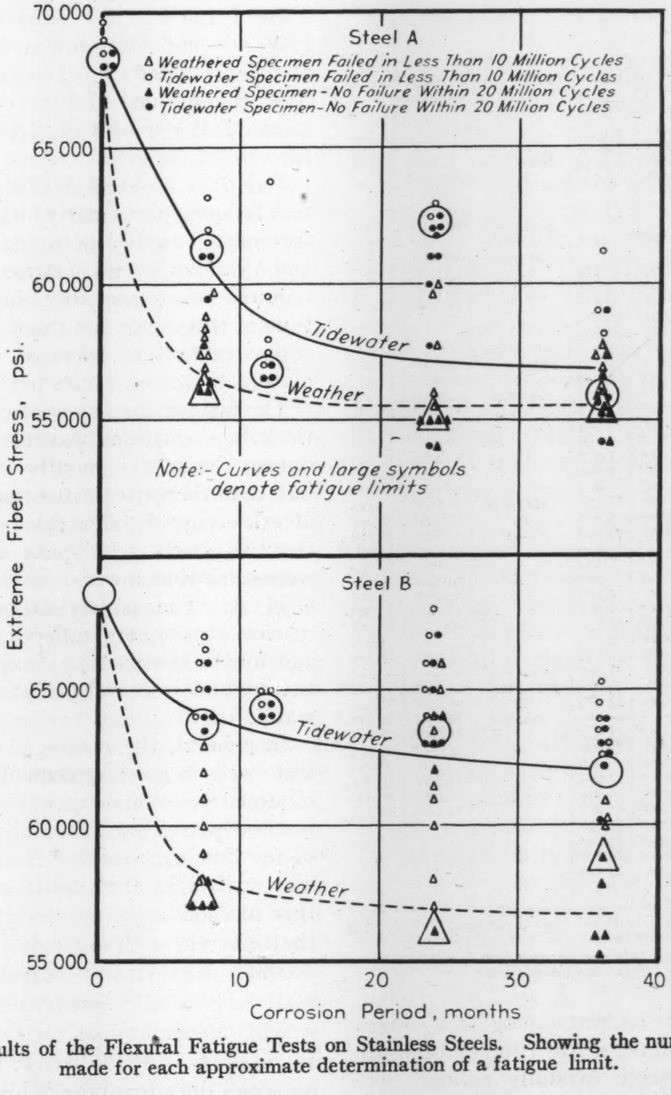

FIG. 5.—Results of the Flexural Fatigue Tests on Stainless Steels. Showing the number of tests made for each approximate determination of a fatigue limit.

A few experiments were also made to discover the approximate distribution of the stresses on the flexed specimens. Samples were coated with a brittle lacquer commercially designated "Stresscote,"[6] and flexed until cracks appeared in the coating. The tests revealed (Fig. 4) that the stresses were fairly uniform over the reduced sections of the specimens, and highest along the edges. Prior to all tests the cut edges of the specimens were carefully rubbed with Aloxite paper, until no burrs were detectable by touch, and until the edges were very slightly rounded.

TABLE I.—RESULTS OF FLEXURAL FATIGUE TESTS ON STAINLESS STEEL[a] PANELS EXPOSED TO TIDEWATER AND TO THE WEATHER.

Exposure	Extreme Fiber Stress,[b] psi.	Number of Cycles $\times 10^3$	Exposure	Extreme Fiber Stress,[b] psi.	Number of Cycles $\times 10^3$
None uncorroded			Tidewater —7½ months	63 200*	1 220
	69 700*	828		62 000*	1 230
				61 500*	1 200
	69 000*	740		61 000[c]	15 280
	68 500*	1 145		61 000	20 700
	68 500	12 450		59 500	10 380
	68 000[c]	15 000			
	68 000	15 300			
Weather—7½ months	59 700*	1 726	Tidewater —12 months	63 800*	750
	58 800*	1 810		59 600*	651
	58 300*	1 388		58 000	962
	58 000*	1 612		57 500*	3 192
	57 800	12 684		57 000*	1 870
	57 500*	828		57 000	12 710
	57 500*	1 902		56 500[c]	17 800
	57 000*	8 918		56 500	43 400
	56 500*	473			
	56 500	12 636	Tidewater —24 months	63 000*	799
	56 000[c]	10 240		62 500*	1 198
	56 000	28 600		62 500	20 810
				62 000[c]	13 000
Weather—24 months	60 000*	624		62 000	22 983
	59 700*	980		61 800	20 724
	57 700*	4 000		61 000	15 910
	56 000*	806		61 000	26 000
	55 500*	16 632		60 000	10 302
	55 000[c]	20 473		57 700	12 600
	55 000	20 693		54 000	18 535
	55 000	21 140			
	55 000	23 021	Tidewater —36 months	61 200*	992
	54 000	12 690		59 000*	1 198
				59 000	24 894
Weather—36 months	57 750*	1 412		58 200*	1 293
	57 400	25 620		56 000*	1 265
	57 400*	966		55 800	20 000
	56 800*	2 304		55 500[c]	20 289
	56 750	20 026		55 200	20 030
	56 200*	1 373		54 150	20 249
	55 850	23 018			
	55 300[c]	20 346			
	55 300	20 597			
	54 000	20 423			

[a] Steel A, with 19.99 per cent chromium, 9.82 per cent nickel, 0.09 per cent carbon.

[b] Asterisks indicate specimens which failed.

[c] Value selected for the fatigue limit.

The results of the fatigue tests on two steels, designated A and B, are given in Table I and FIG. 5. Steel A contained 19.99 per cent of chromium, 9.82 per cent of nickel, and 0.09 per cent of carbon. Steel B contained 17.5 per cent of chromium, 7.35 per cent of nickel, and 0.11 per cent of carbon.

The data for steel A (Table I) reveal that failures, particularly on the corroded specimens, usually occurred between $\frac{1}{2}$ and $1\frac{1}{2}$ million cycles of stress. The time required was reasonably short, for this type of test, since the three Krouse machines each were operated at approximately $2\frac{1}{2}$ million cycles per 24 hr.

The fatigue tests showed (Fig. 5) that the rate of corrosion was relatively rapid during the first 6 months of exposure, but that thereafter a near arrest in the attack occurred. The rates of attack on the two steels were quite similar, but were somewhat lower on steel B than on steel A. The panels exposed to the marine atmosphere suffered more damage, for the same period of exposure, than those which were wet periodically by sea water.

In general, the results of the fatigue tests were in good agreement with those obtained by visual inspection, and the method permitted a reliable evaluation of the damage resulting from corrosion. The scatter in the results obtained by this method appeared no greater than that inherent with other commonly used methods of evaluating corrosion damage, such as visual examinations, loss in weight determinations, or tension tests. In common with the latter, the method possesses the advantage of furnishing the structural engineer with data related to the physical properties in which he is particularly interested.

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[6] A. V. de Forest and Greer Ellis, "Brittle Lacquers as an Aid to Stress Analysis," *Journal of the Aeronautical Sciences*, Vol. 7, No. 5, p. 205 (1940).

DISCUSSION

Mr. F. L. LaQue.[1]—The authors are to be commended for exploring another method of measuring corrosion damage which should be particularly useful with the type of material under consideration, especially after atmospheric exposure where changes in appearance and weight loss determinations frequently fail to disclose or measure real differences in behavior.

It is not evident from the paper that the distribution of corrosion created any difficulty in choosing how or where to cut the specimens for the fatigue tests. It would be anticipated that in many cases, and particularly in connection with specimens immersed in salt water, the tendency for corrosion to be highly localized or to show some peculiar orientation relative to the direction of rolling would result in a considerable variation in the fatigue test results, depending on the choice of specimen position in respect to the nature and location of the attack.

This problem is, of course, common to all methods of measuring corrosion which involve cutting relatively large pieces into specimens for mechanical tests after exposure. Perhaps it would be in order to consider the exposure of test specimens of the approximate shape to be used in the final fatigue tests, making the specimens just enough oversize so as to enable the exposed edges to be machined off and thus to reduce, if not eliminate, the effects of edge attack which would be particularly significant in the case of fatigue test specimens. Such specimens could be made so that any effects of the direction of rolling could be observed.

It seems desirable to emphasize that the results of the present tests are not necessarily indicative of the general behavior of the alloys studied in the types of environment in which these tests were made. Observations by the writer on similar alloys in marine atmospheres and in sea water support the following conclusions which are contrary to the results of the present investigation:

1. Alloys of the type of steel A (19.99 per cent chromium, 9.82 per cent nickel, 0.09 per cent carbon) are generally superior to those of type B (17.5 per cent chromium, 7.35 per cent nickel, 0.11 per cent carbon) in resisting attack both by marine atmospheres and salt water.
2. Attack of these alloys by marine atmospheres is much less severe than by salt water.
3. Attack by salt water generally does not decrease with time.

These apparent contradictions in conclusions are believed to be due primarily to some peculiarity of the salt water test location used in the present investigation rather than to any differences between fatigue tests and other methods of appraising corrosion damage.

Mr. William J. Mayer.[2]—If reference is made to the report of the Materials Corrosion Investigation at Eastport, Me., by the Corps of Engineers of the U. S. Army, photographs of tests specimens will be found. It will be noted that the corrosion of "stainless steels" of the 300 series seems to follow a definite pattern. These specimens were installed in 1935 or 1936 and last year at the inspection period I had the opportunity to examine them.

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[1] Metallurgist, Development and Research Division, The International Nickel Co., Inc., New York, N. Y.

[2] Manager, Engineering Service Dept., A. M. Byers Co., Pittsburgh, Pa.

Corrosion of the stainless plates seems to have followed the direction of rolling of the metal and had the same general appearance as wood beneath the bark of a tree where worms have furrowed parallel with the wood grain.

I wonder whether this peculiar corrosive attack has been taken into consideration when the author's corrosion-fatigue tests were run on the strips. The direction of rolling of the material apparently should be considered. Unless this has been considered in the test, I cannot see how one system can be compared with another.

MR. W. N. FINDLEY.[3]—There are three questions I would like to raise in connection with the application of the fixed cantilever type of fatigue machine for testing thin sheet, in which the specimen is deflected a given amount in each cycle. These are questions which have been troubling the writer and others engaged in the formulation of specifications for the fatigue testing of plastics.[4]

(1) What is the effect of the unbalanced inertia force from the wrist-pin clamp on the stresses developed in such a thin specimen?

T. T. Oberg, and R. D. Lowry, have each studied the effect on the specimen of the inertia of the wrist-pin clamp. Both of these investigators have used essentially the same machine and specimen as the author, and have found it necessary to replace the type of wrist-pin clamp, shown in the paper, by a split wrist pin so that no additional load is applied to the specimen itself as a result of inertia of the wrist-pin clamp.

Another possible solution might be to so shape the wrist-pin clamp that the center of percussion of the clamp is located at the wrist pin.

(2) Can we assume that the equation $S = Mc/I$ gives the maximum stress in the specimen with sufficient accuracy when the specimen is a thin sheet such as used in the tests by the author? Consider that thin sheets must be bent to quite an appreciable curvature in order to obtain stresses which will cause failure in fatigue.

Under such conditions of curvature is the flexure formula, $S = \frac{Mc}{I}$, which is precise only for straight beams, sufficiently accurate for this work? Also when it is considered that the flexure formula is precise only for beams whose width is about the same order of magnitude as the depth, can we assume that the flexure formula gives sufficiently accurate results for specimens of the shape used by the author? Bending of wide beams tends to produce either anticlastic curvature or a biaxial state of principal stress, depending on the relative length and end conditions of the beam.

(3) Was the weight of the wrist-pin clamp considered in calibrating the specimen and setting up the machine? If it was not, the range of stress of the stress cycle would not be complete reversal. That is, the mean stress of the cycle would not be zero, as assumed, but perhaps as much as 5 or 6 per cent of the alternating component of the cycle.

None of the points raised in this discussion would seriously affect the comparative results of corrosion tests. They may, however, affect the numerical results of the fatigue tests.

MESSRS. W. H. MUTCHLER[5] **and J. A. KIES**[5] (*authors' closure*).—As Mr. LaQue has inferred, the problems of selecting samples from corroded panels, and of choosing the method for evaluating damage, are closely related. If, for example, corrosion is very severe, but restricted or directional in its distribution over the surface, methods which aim to evaluate damage by determinations of mechanical properties are generally precluded.

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[3] Instructor in Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[4] Tentative Method of Repeated Flexural Stress (Fatigue) Test of Plastics (D 671 – 42 T), 1942 Book of A.S.T.M. Standards, Part III, p. 1251.

[5] Associate Metallurgist, National Bureau of Standards, Washington, D. C.

Since, on the panels reported upon in this paper, the distribution of the corrosion was relatively uniform, the sampling presented no difficulties. Excluding a $1\frac{1}{2}$ in. length at the end of each panel, the remainder was regularly subdivided into 12 rectangles approximately $2\frac{1}{4}$ by $1\frac{3}{8}$ in., and specimens longitudinal to the direction of rolling were machined from each for fatigue testing.

Directional corrosion may, at times, be owing to the manner in which panels are suspended, instead of resulting from nonhomogeneities in the metal. It is therefore often advisable to expose large samples to the corroding medium, with a view to discarding the ends on which such corrosion is the most likely to be confined.

Reference to Fig. 5 reveals that the fatigue limits of the two steels, for a given period of exposure, differed within a relatively narrow range of ±1000 to 3500 psi. Hence, on the basis of these tests, the conclusion most deserving of emphasis is that they corroded so similarly that, for most service applications, they would prove equally satisfactory. With alloys so closely related in chemical composition it is not unusual because of uncontrolled variables, to find that one or the other may appear slightly the more resistant to attack in separate corrosion tests presumably performed under identical conditions.

The more severe attack on panels exposed to the atmosphere was owing, in part, to abnormal depositions of soot from nearby locomotives. However, the fact that the rate of corrosive attack may decrease with time, and in salt solutions, has been established. This phenomenon was noted by Dix,[6] in a paper presented before this Society in 1933, and has been confirmed in many instances in tests made at the National Bureau of Standards.

The errors introduced by ignoring the angular and translational accelerations of the wrist pin clamp, concerning which Mr. Findley inquires, were determined to be less than one-half per cent. Among other possible sources of error which might be mentioned are the forces due to the accelerations in the specimen itself, the friction in the wrist pin, the air friction, limitations of accuracy in settings and alignments, corner and fillet effects, and variations in room temperature. However, stroboscopic observations revealed no significant secondary deflection due to clamp inertia.

No particular claims are made for accuracy in the determination of the fatigue limit by this method, so that errors of the type listed and theoretical considerations as to formulas involved are relatively unimportant. The important points are that the method affords reproducible results, and, by establishing the approximate fatigue limits of corroded samples, permits the determination of comparative rates of attack between alloys of related chemical composition.

The setting of the mean stress at zero was accomplished by means of a special jig designed by Mr. Kies. The calibration of each specimen was done in a manner such that the weight of the clamp had no effect upon the setting of the mean stress. A description of the method will be furnished gladly upon request.

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[6] E. H. Dix, Jr., "Corrosion Resistance of Structural Aluminum," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 405 (1933).

THE INFLUENCE OF THE COMBINATION OF PRINCIPAL STRESSES IN FATIGUE OF METALS

By D. J. McAdam, Jr.[1]

Synopsis

In recent papers by the author, evidence has been presented that the technical cohesive strength of a metal cannot be represented by a single stress value, but comprises an infinite number of values depending on the combination of the principal stresses. This paper discusses the relation between the three-dimensional diagram representing the influence of the combination of principal stresses on the technical cohesion limit and yield stress, and diagrams representing results of various investigations of the influence of the combination of principal stresses on the fatigue limit. "Chafing fatigue" is considered to be a manifestation of the influence of combined radial compression and cyclic flexure. The damaging effects of combined radial compression and cyclic flexure have been interpreted with reference to diagrams representing the influence of the combination of principal stresses on the technical cohesion limit.

According to the prevalent view, the technical cohesive strength of a metal, in any particular state as regards prior mechanical treatment and heat treatment, may be represented by a single value of the algebraically greatest principal stress.[2] Opposing views, however, have been expressed by some investigators, notably Bridgman (1–4).[3] As a result of compression tests, Bridgman reached the conclusion that the technical cohesion limit varies with the combination of principal stresses. In two recent papers (20, 21), the author has expressed similar views based on published results of tension tests of notched specimens, and has presented two-dimensional diagrams and projections of three-dimensional diagrams showing qualitatively the variation of the technical cohesion limit, ultimate stress, and yield stress with the combination of principal stresses.

By a technical cohesion limit is meant the mean stress at actual or potential[4] rupture. An example of a technical cohesion limit is the "true" breaking stress of a tension test specimen, the breaking load divided by the sectional area at fracture. When a metal is ductile, however, the cohesion limit thus determined has been greatly altered by the plastic deformation during the test. According to a prevalent view, plastic extension first increases, then decreases the technical cohesive strength of a metal, and fracture occurs when the continuously rising curve of flow stress[5] intersects the descending curve of technical cohesion. As shown in two previous papers by the author (20, 21), however, plastic extension causes a continuous increase in the technical cohesive strength, but the increase of the flow stress is faster. The intersection of a curve of flow stress and the corresponding curve of cohesion is at a small angle.

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[1] Metallurgist, National Bureau of Standards, Washington D. C.

[2] Any stress system may be resolved into three principal stresses normal to three mutually perpendicular planes, known as the "principal planes," on which there is no shearing stress. Tensile stresses are viewed as positive, and compressive stresses as negative.

[3] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 591.

[4] Because plastic deformation increases the cohesive strength, the initial cohesion limit of a ductile metal sometimes can be only estimated, not actually attained.

According to the generally accepted theory, the yield strength of metal may be represented by a constant value of the shearing strain energy, and hence by a constant value of the sum of the squares of the principal stress differences. In one of the previous papers (21), however, the author presented evidence that the yield stress depends also on the volume stress, the algebraic average of the three principal stresses.[6]

The failure of a metal by fatigue must be attributed to a progressive failure of cohesion, in the region where the flow stress first reaches the corresponding value of the cohesive strength. (Because of the stress gradient and deformation gradient, however, this local stress value cannot be considered a *technical* cohesion limit.) The nominal fatigue limit can generally be correlated more nearly with the ultimate tensile strength, or with some other index of resistance to plastic deformation, than with an index of technical cohesive strength. Gough and Pollard (10–12) compared the results of their investigation of the influence of the combination of principal stresses on the fatigue limit with various theories of variation of the yield stress. With one steel they found fairly close agreement with the theory of constant maximum shear energy; with other steels they found closer agreement with the theory of constant total energy. These investigators, however, do not suggest that there is any *a priori* reason for such agreement.

Nevertheless, the form of a three-dimensional diagram representing the variation of the fatigue limit with the combination of principal stresses must be influenced directly or indirectly by the form of a corresponding diagram of variation of either the yield stress, ultimate stress, or technical cohesion limit. As the author has presented new two-dimensional and three-dimensional diagrams for the variations of these indices of static strength, it appears desirable to discuss the relation between these diagrams and corresponding diagrams representing the variations of the fatigue limit. It can be shown that some of the discrepancies between the fatigue limits obtained with smooth, unnotched specimens, and the fatigue limits of machinery parts are explainable in terms of the combination of principal stresses.

The Influence of the Combination of Principal Stresses on the Technical Cohesion Limit, Ultimate Strength, and Yield Stress.

The Technical Cohesive Strength of a Brittle Metal:

Only a brief discussion can here be given of the influence of the combination of principal stresses on the technical cohesion limit and resistance to plastic deformation. For detailed discussion, reference may be made to the previous papers on this subject (20, 21). An illustration from one of the previous papers (21) is shown in Fig. 1. Figure 1 (*a*) is a two-dimensional representation of the technical cohesive strength of a

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[5] By "flow stress" is meant the yield stress based on the corresponding sectional area, not on the original area.

[6] Any stress system may be resolved into a polarsymmetric stress (a term used by Kuntze) causing pure volume-strain, and a combination of pure shearing stresses. By "polarsymmetric stress" is meant a system consisting of three equal principal stresses.

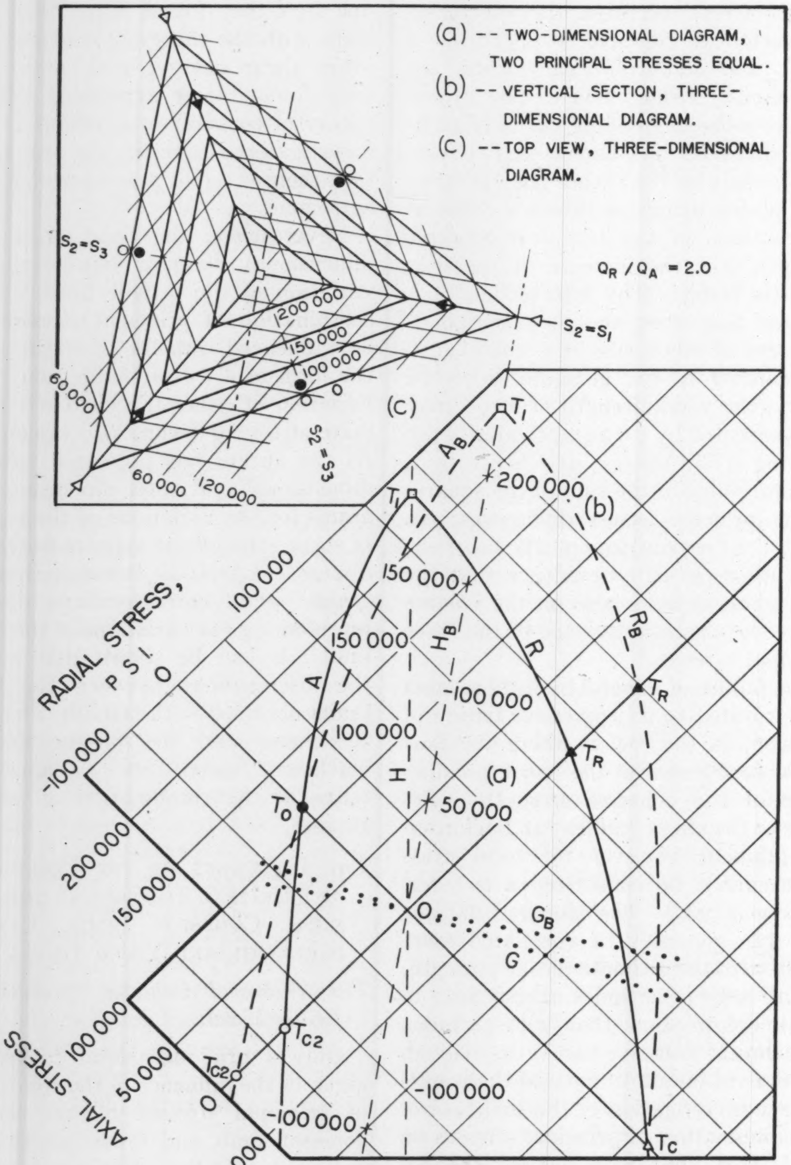
(a) -- TWO-DIMENSIONAL DIAGRAM, TWO PRINCIPAL STRESSES EQUAL.
(b) -- VERTICAL SECTION, THREE-DIMENSIONAL DIAGRAM.
(c) -- TOP VIEW, THREE-DIMENSIONAL DIAGRAM.
$Q_R / Q_A = 2.0$
$S_2 = S_3$
$S_2 = S_1$
$S_2 = S_3$
200 000
150 000
100 000
0
60 000
60 000
120 000
(c)
T_1
A_B
200 000
(b)
T_1
150 000
H_B
R_B
R
150 000
A
-100 000
T_R
T_R
100 000
H
(a)
50 000
T_0
O
G_B
G
T_{C2}
T_{C2}
-100 000
-100 000
T_C
RADIAL STRESS, P S I.
100 000
0
-100 000
200 000
150 000
100 000
50 000
0
-50 000
AXIAL STRESS

Fig. 1.—Top View and Vertical Section of Three-Dimensional Diagram Representing Technical Cohesive Strength of Brittle Steel.

brittle metal. Figure 1 (*b*) and (*c*) represent a vertical section and a top view, respectively, of a three-dimensional diagram.

Figure 1 (*a*) represents stress combinations with two of the principal stresses equal.[7] Stress combinations of this kind would be produced by subjecting a cylinder to axial stress and uniform radial stress. The uniform radial stress may be resolved into two equal principal stresses, whose mutually perpendicular directions are rotatable around the specimen axis. In such a system the stress is the same in any direction in a plane perpendicular to the axis. Stress combinations with two of the principal stresses equal are of two types. In one type, the axial stress is algebraically greater than the radial stress; the axial stress thus is S_1, and S_2 is equal to S_3. Combinations of this type are represented by curve A of Fig. 1 (*a*). In the other type, the radial stress is greater than the axial stress; the radial stress then is S_1, and S_2 is equal to S_1. Combinations of this type are represented by curve R of Fig. 1 (*a*).

Line H, making an angle of 45 deg. with the axes of coordinates, is the locus of all points representing polarsymmetric[6] stress ($S_1 = S_2 = S_3$). The point T_1 represents the "disruptive stress," the polarsymmetric stress necessary to cause rupture. The points designated T_o and T_c represent rupture under unidirectional tension and compression, respectively. The points T_{c2} and T_R represent rupture under radial compression and tension, respectively. Line G is the locus of points representing pure shear, and the intersections of this line with curves A and R represent rupture under pure shearing stress. (The radial stress is arithmetically half the longitudinal stress.)

Another curve of this type is curve A of Fig. 6 (*a*). The radiating lines represent constant values of the ratio (S_3/S_1) of radial to axial stress. The intersections of these lines with curve A represent corresponding values of the technical cohesion limit. As the technical cohesive strength of a metal cannot be represented by a single stress value, any specific cohesion limit must be referred to the corresponding combination of the principal stresses. When S_2 is equal to S_3, any cohesion limit may be designated with reference to the corresponding value of S_3/S_1. The cohesion limits for unidirectional and polarsymmetric stress, therefore, have been designated T_o and T_1, respectively, and the cohesion limit corresponding to any other value of S_3/S_1 has been designated by T with this value as a subscript.

Removal of the restriction that at least two of the principal stresses must be equal makes it necessary to represent the technical cohesive strength of a metal by a three-dimensional diagram. A complete three-dimensional diagram has three equivalent, mutually perpendicular axes of unidirectional stress, three equivalent planes representing zero value of one of the principal stresses, and three equivalent planes (120 deg. apart) representing stress combinations with two of the principal stresses equal. The surface of the diagram represents all possible combinations of the principal stresses at the technical cohesion limit; there are generally six equivalent points for any stress combination. The locus of polarsymmetric stresses makes equal angles with the directions of the three principal stresses, and is symmetrically situated with reference to the three equivalent axes of unidirectional stress. As the locus of polarsymmetric stresses

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[7] In this paper, as in the two previous papers (20, 21) the algebraically greatest principal stress will be designated S_1, the least principal stress will be designated S_3, and the intermediate principal stress will be designated S_2.

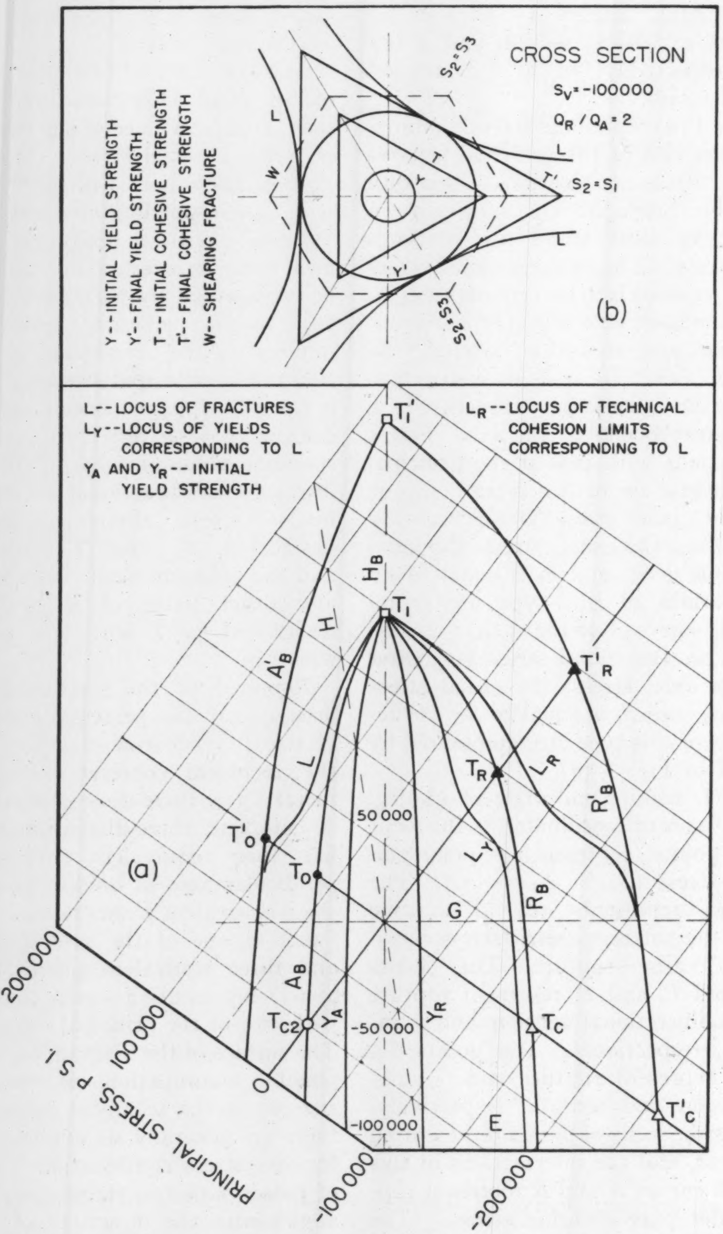
CROSS SECTION
S_V = -100000
Q_R / Q_A = 2
$S_2 = S_3$
$S_2 = S_1$
$S_2 = S_3$
L
L
L
W
T
T'
Y
Y'
(b)
Y--INITIAL YIELD STRENGTH
Y'--FINAL YIELD STRENGTH
T--INITIAL COHESIVE STRENGTH
T'--FINAL COHESIVE STRENGTH
W--SHEARING FRACTURE
L--LOCUS OF FRACTURES
L_Y--LOCUS OF YIELDS CORRESPONDING TO L
Y_A AND Y_R--INITIAL YIELD STRENGTH
L_R--LOCUS OF TECHNICAL COHESION LIMITS CORRESPONDING TO L
T'_I
T_I
H_B
H
A'_B
L
T'_R
T_R
L_R
R'_B
L_Y
T'_O
T_O
(a)
50 000
G
R_B
A_B
T_{C2}
Y_A
-50 000
Y_R
T_C
T'_C
-100 000
E
200 000
100 000
0
-100 000
-200 000
PRINCIPAL STRESS, P S I

Fig. 2.—Vertical and Cross-Sections of Three-Dimensional Diagram Representing Technical Cohesive Strength and Yield Strength of Ductile Steel.

is an axis of symmetry of the three-dimensional diagram, the form of the diagram may be clearly revealed by two views; a view of a section containing the axis of symmetry and representing stress combinations with two of the principal stresses equal, and a (top) view in the direction of this axis. A vertical section of such a diagram is represented by Fig. 1 (*b*) and a top view is represented by Fig. 1 (*c*).

In a three-dimensional diagram, the angle between the locus of polarsymmetric stresses (H) and the directions of each of the principal stresses is $\tan^{-1}\sqrt{2}$, about 54 deg. 44 min. Any point in Fig. 1 (*b*) has the same ordinate as the corresponding point in Fig. 1 (*a*) but the abscissa of a point in Fig. 1 (*b*) is derived by multiplying the corresponding abscissa in Fig. 1 (*a*) by $\sqrt{2}$. Because of this relationship, the line representing pure shear is tilted from the position represented by line G to that represented by line G_B. Line G_B thus is perpendicular to the locus of polar-symmetric stresses H_B.

In a three-dimensional diagram, there are three sections representing stress combinations with at least two of the principal stresses equal. These sections pass through the axis of symmetry and are 120 deg. apart. In Fig. 1 (*c*), the top view, these sections appear as three lines radiating from the axis of symmetry. These lines are cut by contour lines, each corresponding to the indicated value of the volume-stress.[8] The number adjacent to each contour line indicates the corresponding value of the volume stress, which is indicated also on line H_B of Fig. 1 (*b*). The size of the triangular cross-section of the three-dimensional diagram decreases with increase in the volume stress; the diagram thus tapers nonlinearly to the point (T_1) representing the disruptive stress. All points derived from curve A_B are on the sloping sides of the three-dimensional diagram; all points derived from curve R_B are on the sloping edges.

As shown in a previous paper (21), the three-dimensional diagram has either a triangular contour (Fig. 1) or a three-cornered contour with curved sides (Fig. 18 (*a*) of a previous paper (21)). The curvature of the sides possibly increases with increase in the volume-stress.

The Technical Cohesive Strength, Ultimate Strength, and Yield Strength of a Ductile Metal:

A three-dimensional diagram representing the initial technical cohesive strength of a ductile metal, or of a metal at any instant during plastic deformation, probably is qualitatively similar to the three-dimensional diagram for a brittle metal (Fig. 1). Figure 2 (*a*) shows a vertical section ($S_2 = S_3$) of a three-dimensional diagram representing the technical cohesive strength and yield strength of a ductile metal. Curves A_B and R_B (similar to the curves so designated in Fig. 1 (*b*)) represent the *initial* technical cohesive strength. Except at the point (T_1) representing the disruptive stress, these curves are outside the curves (Y_A and Y_B) representing yield strength. Any combination of sufficiently high stresses excepting polar-symmetric stress, therefore, would cause plastic deformation, and thus would increase the size of the three-dimensional diagram representing the technical cohesive strength. After considerable deformation, the vertical section would be represented by curves A'_B and R'_B. Curve L is the locus of fractures, when S_2 is equal to S_3. Its complex form is due to the influence of two variables, plastic deformation and S_3/S_1.

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[8] The volume-stress is equal to the algebraic average of the three principal stresses. It thus is $(S_1 + S_2 + S_3)/3$.

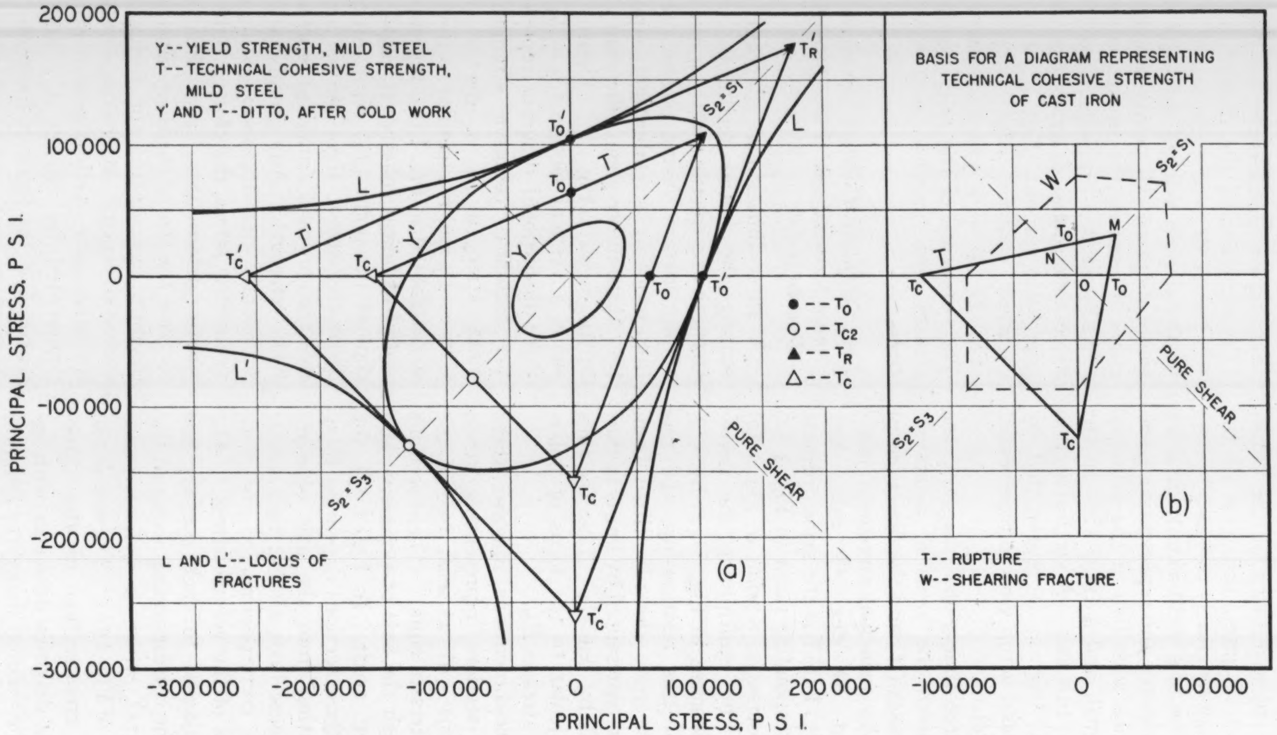

Fig. 3.—Oblique Section of Three-Dimensional Diagram Representing Technical Cohesive Strength of Steel, and Basis for Diagram for Gray Cast Iron. Section at zero value of one of the principal stresses.

Curves representing the ultimate strength would be similar in form to the curves representing the technical cohesive strength and yield strength.

Figure 2 (*b*) represents a cross-section at a volume-stress of minus 100,000 psi., in the position of line E of Fig. 2 (*a*). The initial and final technical cohesive strengths are represented by triangles T and T', respectively; the initial and final yield strengths are represented by circles Y and Y', respectively. The three-dimensional diagram representing yield strength has a circular cross-section, and tapers nonlinearly to the point representing the disruptive stress. With plastic deformation, this diagram increases in size, and the circular cross-section eventually touches the middles of the sides of the increasing triangles representing the technical cohesive strength (Fig. 2 (*a*)). Rupture then occurs if S_2 equals S_3, but additional plastic deformation is possible if S_2 is greater than S_3. Unlimited plastic deformation of a very ductile metal is possible when S_2 is nearly equal to S_1 (and when S_3/S_1 is not too high). This relationship is illustrated by the discontinuities in the locus of rupture (L) in Fig. 2 (*b*).

The diagram representing fracture by shear may have the hexagonal cross-section represented by W in Fig. 2 (*b*). It is also possible that the cross-section is circular. In either case, the diagram may intersect the diagram for rupture as indicated qualitatively in the figure.

Each of three equivalent sections of the three-dimensional diagram represents stress combinations with zero value of one of the principal stresses. One of these sections is perpendicular to the plane of Fig. 2 (*a*), and passes through the axis of abscissas, the line extending through T_{c2} and T_R. A view of such a section is shown in Fig. 3 (*a*). In this oblique section of the three-dimensional diagram, the triangles T and T', the approximately elliptical sections Y and Y', and the locus of fractures L, have the same designations as in the cross-sectional view, Fig. 2 (*b*). The sections representing yield strength are not ellipses in the oblique section, because the three-dimensional diagram representing yield strength tapers with increase in the volume stress.

The Technical Cohesive Strength of Gray Cast Iron:

As fatigue of gray cast iron under various stress combinations is to be discussed later, a brief discussion will now be given of the probable form of the diagram representing the technical cohesive strength of this material.

Under some stress combinations, gray cast iron exhibits considerable plasticity. Moreover, fracture under some stress combinations is by shear rather than by rupture. A complete diagram representing the technical cohesive strength of gray cast iron, therefore, is of complex form. The initial technical cohesive strength, for resistance to rupture, may be represented in oblique section by triangle T of Fig. 3 (*b*), although the sides of the contour possibly are curved. The fact that the shearing strength of cast iron is greater than its tensile strength may be due largely to the plastic deformation possible under pure shear and may not mean that the section representing the *initial* technical cohesive strength has a greater ordinate at N than at M. As a shearing fracture occurs under unidirectional compression, the section (W) representing shearing fracture must cross the axis of abscissas between 0 and T_c as indicated in the figure.

EFFECT OF COMBINING CYCLIC STRESS WITH STATIC STRESS OF A DIFFERENT KIND

That the influence of static tension on the torsional fatigue limit is of con-

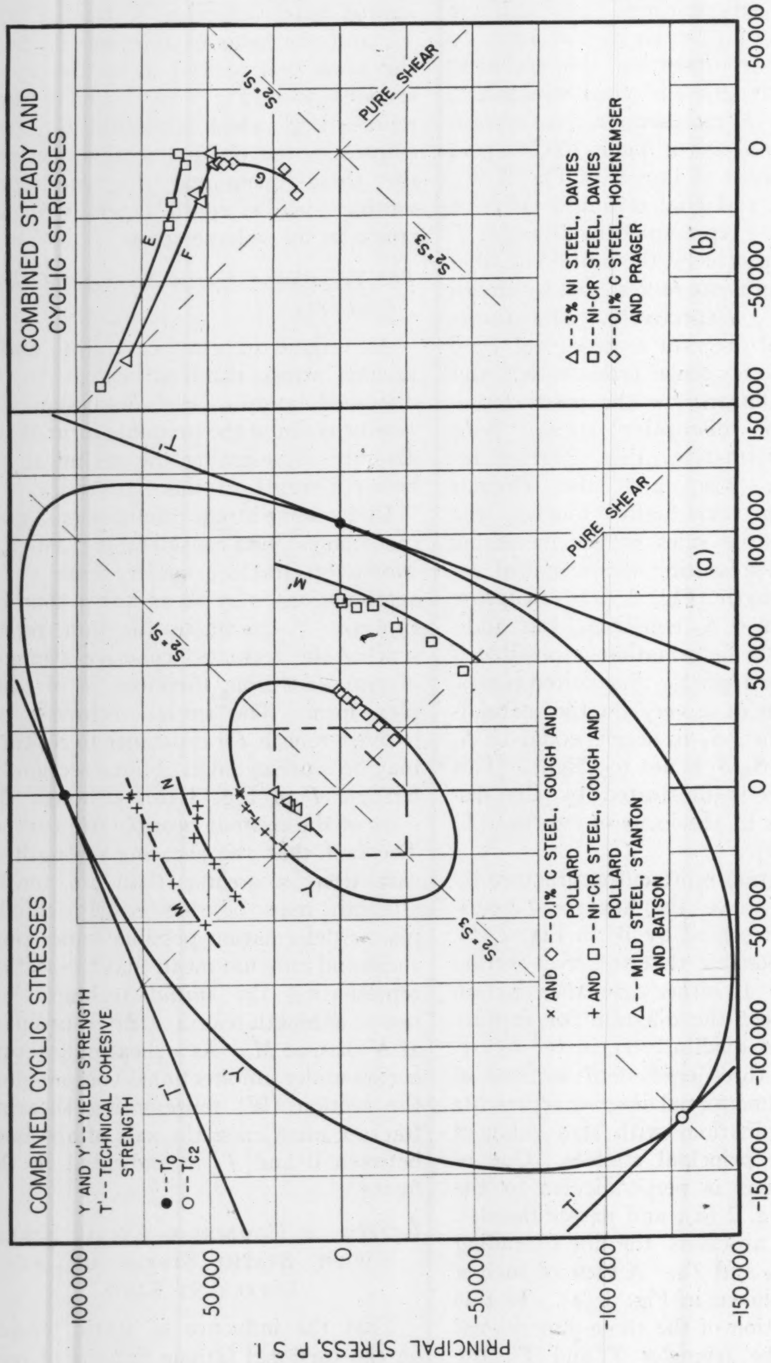

FIG. 4.—Oblique Section of Three-Dimensional Diagram for Technical Cohesive Strength Compared with Curves Representing Influence of Combination of Principal Stresses on Fatigue Limit.

siderable practical importance is indicated by the improvement of the fatigue limit due to relief of longitudinal internal tensile stress. This evidence is supported by results of an investigation by Hohenemser and Prager (**14**) on the quantitative influence of static tensile stress on the fatigue limit for alternating torsion. The torsional fatigue limit was found to decrease at an increasing rate with increase in the longitudinal tensile stress. The equation suggested by Hohenemser and Prager to represent the relationship is

$$q^2/q_1^2 + P^2/P_1^2 = 1$$

where q = the torsional fatigue limit under any given stress combination,
q_1 = the fatigue limit for pure alternating torsion,
P = the applied static tensile stress, and
P_1 = the ultimate tensile stress.

This is the equation for an ellipse, of which P_1 and q_1 are the semiaxes.

In Fig. 4 (*b*), the results are represented in terms of the principal stresses (one of the principal stresses, the radial stress in the specimen, is practically zero). Pure torsional fatigue is represented by the lower end of curve *G*, and the course of this curve shows the influence of increasing static tensile stress.

The influence of static torsion on the flexural fatigue limit is also of much practical importance. Such a combination of stresses occurs in many shafts. This subject has been investigated by Ono, discussion of paper by Gough and Pollard (**10**), and by Lea and Budgen (**19**). Both investigations led to the same conclusion, that static torsional stress does not diminish the flexural fatigue limit appreciably unless the torsional stress exceeds the half range of the flexural stress. Above a certain value of the torsional stress, however, there is an abrupt decrease in the flexural fatigue limit.

In discussing a paper by Gough and Pollard (**10**), Ono reported that cast iron under combined static torsion and alternating flexure behaves differently from steel. The fatigue limit of the cast iron was found to be considerably affected by static torsion. The investigation, however, had not been completed.

Davies, in discussing the same paper by Gough and Pollard (**10**), said that the influence of static torsion on the flexural fatigue limit had been studied for 5 or 6 yr. at Battersea Polytechnic Institute and had been discussed in theses for the degree of master of science. The results given by Davies are represented by curves *E* and *F* of Fig. 4 (*b*). The fatigue limits under pure flexure are represented by the lower ends of these curves, and the course of each curve shows the influence of increasing static torsional stress. The results are not in agreement with those of Ono and of Lea and Budgen (**19**). Static torsion, even in relatively small amount, evidently decreased the flexural fatigue limit. The equation suggested by Davies was (with change of letters)

$$P^2/P_1^2 + Q^2/Q_1^2 = 1$$

where P = the flexural fatigue limit (semirange) for any stress combination,
Q = the corresponding static torsional stress,
P_1 = the fatigue limit for pure alternating flexure, and
Q_1 = the static ultimate torsional stress.

This is the equation for an ellipse, of which P_1 and Q_1 are the semiaxes.

Much further investigation of this subject evidently is needed. It should include also combinations with varying mean stress of the cycle as well as varying static stress.

EFFECT OF COMBINING CYCLIC TENSILE (OR BENDING) STRESS WITH CYCLIC TORSIONAL STRESS

All reported investigations of combined cyclic bending and cyclic torsion have been made with the two cycles in phase. The earliest reported investigation was by Stanton and Batson (27). In their experiments, the ratio of the torsional moment to the bending moment ranged from zero to 20. The results, obtained with a mild steel, were said to correspond rather closely with a constant value of the maximum shearing stress.

An extensive investigation of the subject was started at the National Physical Laboratory, and three papers have been published by Gough and Pollard (10–12). Their results were obtained by combining alternating plane flexure with alternating torsion. The materials used were steels and cast irons. Both solid and hollow specimens of two steels were used. The fatigue limits were generally lower for hollow than for solid specimens. The differences were slight (0 to 4 per cent) for pure alternating flexure, but increased to $7\frac{1}{2}$ to 10 per cent for pure alternating torsion. The variation of the fatigue limit with the combination of principal stresses, however, was qualitatively the same for both solid and hollow specimens. As the torsional endurance range is increased from zero, the flexural endurance range decreases. An increase of the flexural endurance range has a similar effect on the torsional endurance range. By plotting as coordinates the corresponding endurance half ranges, Gough and Pollard found each curve to be an ellipse, which may be represented by the equation

$$f^2/f_1^2 + q^2/q_1^2 = 1$$

where f and q = the half ranges of bending and torsional stresses for any given stress combination, and

f_1 and q_1 = the fatigue limits for pure alternating flexure and pure alternating torsion, respectively.

This equation is identical in form with the equations suggested by Hohenemser and Prager (14) and by Davies (10) to represent the effect of combined cyclic and static stresses, differing in kind.

A comparison was made by Gough and Pollard between the variations of the fatigue limit and the criteria of the various theories of the influence of the combination of principal stresses on yield. As a basis for such a comparison, the ratios of the calculated maximum shear stresses (half range) to the value for pure alternating flexure were plotted against the ratios of bending moment to twisting moment. These curves were compared with similar curves drawn in accordance with the various theories. With some metals, the results conformed rather closely to the theory of constant maximum shearing energy; with others, the results were intermediate between the curve representing constant maximum shearing stress and the curve representing constant maximum shear energy.

From the results obtained by Stanton and Batson (27) and by Gough and Pollard (10, 11) calculations have been made of the principal stresses (one of the principal stresses is practically zero), the values obtained have been plotted in Fig. 4 (*a*), and curves have been drawn for qualitative comparison with the boundaries of a section of a three-dimensional diagram representing the technical cohesive strength of a ductile steel. The sectional view here shown is qualitatively similar to the previously described section in Fig. 3 (*a*). Results obtained with solid specimens have been placed above the line of symmetry of this section, and results obtained with hollow specimens have been placed in an equivalent location below this line. Curve M' has been drawn equal to curve M to facilitate comparison.

Although the results obtained by Stanton and Batson conform rather closely to the hexagon representing the criterion for the maximum shear theory, they would conform nearly as well with an ellipse representing the theory of maximum shear energy. The results obtained by Gough and Pollard with the 0.1 per cent carbon steel conform rather well with the approximate ellipse Y. The results obtained with the stronger steels, however, do not conform to an ellipse, but conform rather closely with the shape of the section Y' representing the yield strength of steel that has received considerable plastic deformation. The evidence thus suggests that the form of a curve such as curves M and N of Fig. 4 (*a*) is influenced by the boundary of the three-dimensional diagram representing yield. For a soft steel, this diagram (Fig. 2) is relatively long and slender, and tapers only slowly until it has extended far into the region of three-dimensional tension. For a hard steel, the departure from a nearly cylindrical form is more rapid, and hence an oblique section such as Y' of Fig. 4 (*a*) is further from the elliptical form. Curves M and N, consequently, are further from a true elliptical form than is the curve obtained with the 0.1 per cent carbon steel.

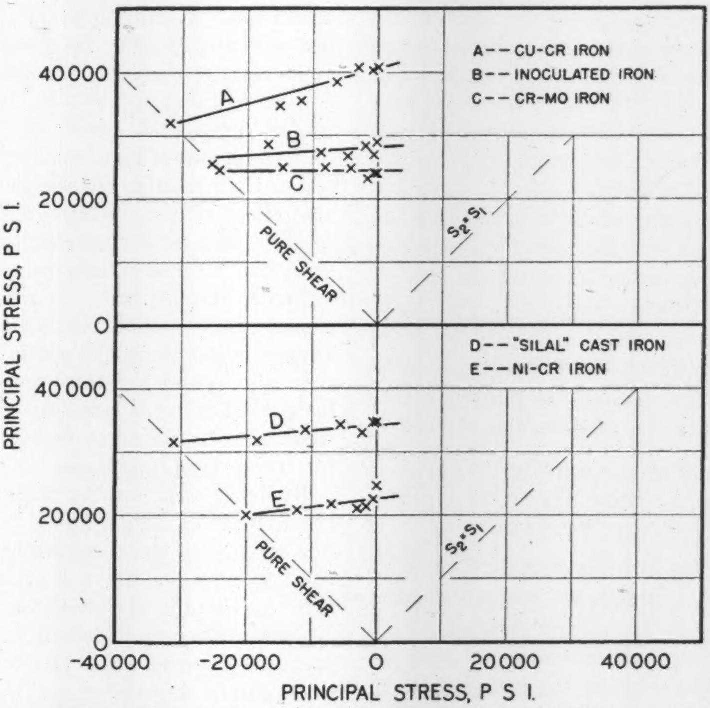

Fig. 5.—Diagrams Representing Influence of Combination of Principal Stresses on Fatigue Limits of Cast Irons.

With cast irons, Gough and Pollard (10, 12) obtained the results represented in Fig. 5, which is of the same type as Figs. 3 and 4. The results evidently may be represented by straight lines. Comparison of these lines with the corresponding side of triangle T of Fig. 3 (*b*), suggests that the variation of the fatigue limit of gray cast iron with the combination of principal stresses is influenced by the adjacent boundary of the three-dimensional diagram representing the technical cohesive strength. The fact that line C of Fig. 5 is nearly horizontal, if considered alone, would suggest, as it did to Gough and Pollard (10), that the fatigue limit of cast iron may be determined by a constant maximum value of the greatest principal stress. The other lines (12) in Fig. 5, however, give no support to this view. The slope of each line probably would be greater if the course were not influenced by the variation of plasticity with the combination of principal stresses.

EFFECT OF COMBINING RADIAL COMPRESSIVE STRESS WITH CYCLIC FLEXURE

In all the stress systems represented in Figs. 3, 4, and 5, the stress normal to the lateral surface of the metal is practically zero. Consideration will now be given to the effect on the fatigue limit of combining cyclic flexure with either steady or repeated lateral compression. Such stress systems are induced in machinery parts by press fits, grips, collets, clamps, bearings, etc. The fatigue limit under such conditions may be much less than that for a smooth specimen free from surface pressure (5, 16–18, 24, 25, 26, 30–32). Even a press-fitted sleeve that transmits no bending moment to the shaft may lower the fatigue limit to less than half the ordinary value. The damaging effect is especially great at the line where the edge of the sleeve exerts repeated pressure on the shaft.[9]

Some investigators of fatigue of press fits have focused attention on the rubbing or chafing action, and the effect has been called "chafing fatigue." Attention has been called to the roughening of the surface and to the fact that such action is associated with the production of fine oxide powder. As shown by Thum and Wunderlich (31), however, the lowering of the fatigue limit has little relation to the amount of this powder formed. Moreover, the fatigue cracks frequently start in relatively smooth regions of high repeated pressure, rather than in adjacent rougher regions. The effect of the surface roughness, of itself, would be no greater than if the shaft had been so roughened prior to an ordinary fatigue test; chafing and repeated flexure could have no conjoint action similar to that of cyclic stress and corrosion. And yet the lowering of the fatigue limit by a press-fitted member frequently is too great to be accounted for by surface roughness.

Evidence will now be presented that the damage caused by such press fits is due largely to the combination of principal stresses. Even the roughening of the surface by the removal of small particles of metal is due to local fatigue under the combination of cyclic flexure and radial compression.

When a cylindrical specimen is subjected to cyclic flexure and to steady or repeated radial compression, the stress system is of the type represented by curve A of Fig. 1 ($S_2 = S_3 < S_1$). A similar curve is shown in Fig. 6 (*a*), and portions of the same curve are reproduced in Fig. 6 (*b*) and (*c*). In each diagram, T_o represents rupture under

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[9] That compressive stress concentration plays an important part in this damage is indicated by the fact that the fatigue limit of a specimen of annealed metal under combined flexural stress and radial compressive stress, may be greater than that of a heat-treated or cold-worked metal. Local yield of the softer metal evidently gives partial relief of the compressive stress concentration.

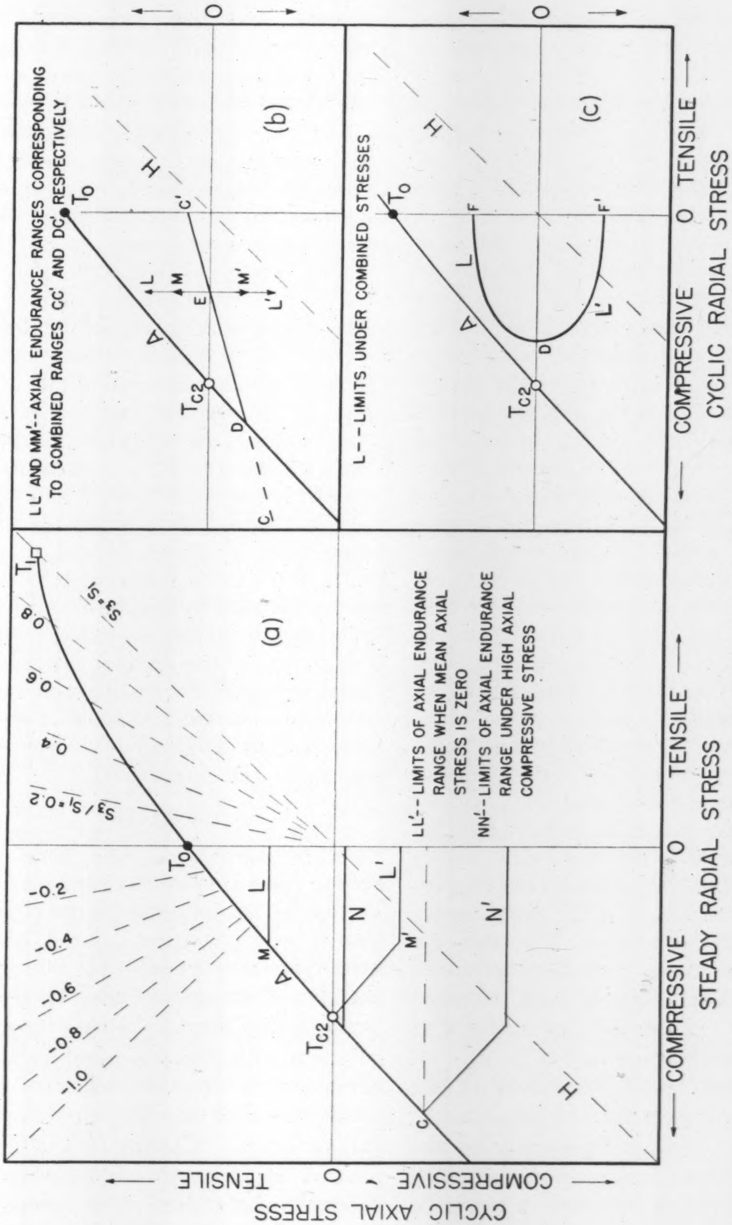
(a)
(b)
(c)
LL' AND MM'-AXIAL ENDURANCE RANGES CORRESPONDING TO COMBINED RANGES CC' AND DC' RESPECTIVELY
L--LIMITS UNDER COMBINED STRESSES
LL'-LIMITS OF AXIAL ENDURANCE RANGE WHEN MEAN AXIAL STRESS IS ZERO
NN'-LIMITS OF AXIAL ENDURANCE RANGE UNDER HIGH AXIAL COMPRESSIVE STRESS
T₀
T₁
T_C2
A
H
L
L'
M
M'
N
N'
C
C'
D
E
F
F'
S₃/S₁=0.2
0.4
0.6
0.8
S₃=S₁
-0.2
-0.4
-0.6
-0.8
-1.0
0
COMPRESSIVE
TENSILE
STEADY RADIAL STRESS
CYCLIC RADIAL STRESS
CYCLIC AXIAL STRESS

FIG. 6.—Influence of Radial Compressive Stress on Fatigue Limit.

static unidirectional tension; T_{c2} represents rupture under radial compression, with no longitudinal stress. As curve A is a boundary of the diagram representing the static technical cohesive strength, this boundary evidently could not be exceeded by any combination of cyclic stresses at the fatigue limit.

If a specimen be subjected to static radial compressive stress of the amount represented by T_{c2} of Fig. 6 (*a*), its flexural fatigue limit evidently would be zero, because the superposition of any flexural stress would push the point representing the stress system beyond curve A. If lines L and L' be assumed to represent the limits of the endurance range, and if it be assumed that this range is not narrowed by the superposition of a static radial compressive stress within the limitations of curve A, decrease of the endurance range would begin when the static radial compressive stress exceeds the value corresponding to point M, and the range would contract as indicated in the figure. A similar variation of the endurance range would occur when the mean flexural stress is compressive, and the limits of the endurance range are lines N and N'.

Many press fits, however, induce *repeated* radial compressive stress superposed on the static radial compressive stress and cyclic flexure. The edge of a sleeve may cause high local repeated pressure. The effect of such repeated pressure is represented qualitatively as a first approximation in Fig. 6 (*b*). If L and L' represent the limits of the endurance range when the mean radial compressive stress is represented by the abscissa of point E, and if the repeated superposed radial compressive stress be represented by the difference between the abscissas of C and E, the limit of the range of compressive stress would be beyond curve A; the fatigue limit under such a stress system, therefore, would be exceeded. At the fatigue limit, the limit of the radial compressive stress could be no greater than that represented by D. The flexural stress range corresponding to this limit evidently would be between M and M'. It is possible thus to account *qualitatively* for the lowering of the flexural fatigue limit because of the superposition of repeated and steady radial compressive stress.

It is probable, however, that the flexural endurance range would be limited, not by the boundary of the diagram representing static technical cohesion, but by a certain nearness to this boundary. Under *repeated* pure radial compressive stress, the fatigue limit probably would be less than that represented by the abscissa of point T_{c2} of Fig. 6 (*c*). If it be assumed that this fatigue limit is represented by point D and that the limits of the stress range for pure flexure are represented by points F and F', the influence of varying repeated compressive stress on the flexural fatigue limit probably would be represented qualitatively by curves L and L'. Further investigation evidently is needed to determine the relationship.

Summary

A brief survey has been made of the recent views presented elsewhere by the author on the influence of the combination of principal stresses on the technical cohesive strength and yield strength of metals. Comparison has then been made of the diagrams representing these relations with data published by various investigators on the influence of the combination of principal stresses on the fatigue limit. "Chafing fatigue" is considered to be a manifestation of the influence of combined radial compression and cyclic flexure. The damaging effects of the radial compression have been interpreted with reference to diagrams representing the influence of the combination of principal stresses on the technical cohesion limit.

REFERENCES

(1) P. W. Bridgman, "Breaking Tests Under Hydrostatic Pressure, and Conditions of Rupture," *Philosophical Magazine,* Vol. 24, July 1912, pp. 63–80.

(2) P. W. Bridgman, "Theoretically Interesting Aspects of High Pressure Phenomena," *Reviews of Modern Physics,* Vol. 7, pp. 1–35 (1935).

(3) P. W. Bridgman, "Reflections on Rupture," *Journal of Applied Physics,* Vol. 9, pp. 517–528 (1938).

(4) P. W. Bridgman, "Considerations on Rupture Under Triaxial Stress," *Mechanical Engineering,* Vol. 61, February 1939, pp. 107–111.

(5) T. V. Buckwalter, O. J. Horger, and W. C. Sanders, "Locomotive Axle Testing," *Transactions,* Am. Soc. Mechanical Engrs., Vol. 60, RR 60-2, pp. 335–345 (1938).

(6) F. W. Caldwell, "Vibration Problems in Aircraft Propeller Designing," *Journal,* Soc. Automotive Engrs., Vol. 41, August 1937, pp. 372–380.

(7) J. H. Frye and G. L. Keeth, "The Fatigue Resistance of Steel as Affected by Some Cleaning Methods," *Transactions,* Am. Soc. Metals, Vol. 26, pp. 192–218 (1938).

(8) H. W. Gillett and E. L. Mack, "Notes on Some Endurance Tests of Metals," *Proceedings,* Am. Soc. Testing Mats., Vol. 24, Part II, p. 476 (1924).

(9) H. J. Gough and W. J. Clenshaw, "The Testing of Engineering Materials," *Transactions,* Inst. Marine Engrs., Vol. 47, Part 10, Section V "Contact Corrosion" (1935).

(10) H. J. Gough and H. V. Pollard, "The Strength of Metals Under Combined Alternating Stresses," *Proceedings,* Inst. Mechanical Engrs., November 1935, pp. 1–103.

(11) H. J. Gough and H. V. Pollard, "The Effect of Specimen Form on the Resistance of Metals to Combined Alternating Stresses," *Proceedings,* Inst. Mechanical Engrs., December 1936, pp. 549–573.

(12) H. J. Gough and H. V. Pollard, "Properties of Some Materials for Cast Crankshafts, with Special Reference to Combined Stresses," *Proceedings,* Inst. Mechanical Engrs., March, 1937, pp. 821–896.

(13) H. Hauttman, "Relation of Laboratory Research to Engine Design," *Transactions,* Northeast Coast. Engrs. and Shipbuilders, Vol. 52, pp. 103–126 (1935–1936).

(14) Hohenemser and Prager, "Zur Frage der der Ermüdungsfestigkeit bei mehrachsigen Spannungszuständen," *Metallwirtschaft,* Vol. 12, 1933, pp. 342–343.

(15) O. J. Horger and T. V. Buckwalter, "Fatigue Strength of 2-in. Diameter Axles with Surfaces Metal-Coated and Flame-Hardened," *Proceedings,* Am. Soc. Testing Mats., Vol. 40, p. 733 (1940).

(16) O. J. Horger, "Effect of Surface Rolling on the Fatigue Strength of Steel," *Transactions,* Am. Soc. Mechanical Engrs., Vol. 57, pp. A 128–136 (1935).

(17) O. J. Horger and J. L. Maulbetsch, "Increasing the Fatigue Strength of Press Fitted Axle Assemblies by Surface Rolling," *Transactions,* Am. Soc. Mechanical Engrs., Vol. 58, pp. A 91–98 (1936).

(18) O. J. Horger and T. V. Buckwalter, "Fatigue-Comparison of 7 inch Diameter Solid and Tubular Axles," *Proceedings,* Am. Soc. Testing Mats., Vol. 41, p. 682 (1941).

(19) F. C. Lea and H. P. Budgen, "Combined Torsional and Repeated Bending Stresses," *Engineering,* Vol. 122, pp. 242–244.

(20) D. J. McAdam, Jr., "The Technical Cohesive Strength of Metals," *Transactions,* Am. Soc. Mechanical Engrs., Vol. 63, *Journal of Applied Mechanics,* December, 1941, Vol. 8, A 155–165.

(21) D. J. McAdam, Jr., "The Technical Cohesive Strength and Yield Strength of Metals," *Technical Publication No. 1414,* Am. Inst. Mining and Metallurgical Engrs., January 1941.

(22) K. Matthaes, *Luftfahrforschung,* Vol. 12, pp. 176–179 (1935).

(23) R. E. Peterson and A. M. Wahl, "Fatigue of Shafts at Fitted Members, with a Related Photoelastic Analysis," *Transactions,* Am. Soc. Mechanical Engrs., Vol. 57, pp. A 1–11 (1935).

(24) "Reiboxydation," Correlated Abstract, *Metallwirtschaft,* Vol. 18, pp. 992–995 (1939).

(25) G. Sachs, "Improving Aircraft Propellers by Surface Rolling," *Metals and Alloys,* Vol. 10, pp. 19–23 (1939).

(26) G. Sachs and P. Stefan, "Chafing Fatigue Strength of Some Metals and Alloys," *Transactions,* Am. Soc. Metals, Vol. 29, pp. 373–399 (1941).

(27) T. E. Stanton and R. G. Batson, "The Fatigue Resistance of Mild Steel Under Various Conditions of Stress Distribution," Report, British Assn., pp. 288–291 (1916).

(28) R. Stribeck, "Dauerfestigkeit von Eisen und Stahl bei wechselnder Biegung, verglichen mit den Ergebnissen des Zugversuchs," *Zeitschrift* des Vereines deutscher Ingenieure, Vol. 67, pp. 631–636 (1923).

(29) A. Thum and E. Wunderlich, "Der Einfluss von Einspann- und Kraftangriffstellen auf die Dauerhaltbarkeit der Konstruktionen," *Zeitschrift* des Vereines deutscher Ingenieure, Vol. 77, pp. 851–853 (1933).

(30) A. Thum and E. Wunderlich, "Dauerfestigkeit von Konstruktionsteilen an Einspannungen, Nabensitzen und aenlichen Kraftangriffsstellen," *Mitteilungen*, Technischen Hochschule, Darmstadt, No. 5 (1934).

(31) A. Thum and E. Wunderlich, "Die Reiboxydation an festen Paarverbindungsstellen und ihre Bedeutung für den Dauerbruch," *Zeitschrift für Metallkunde*, Vol. 27, pp. 277–280 (1935).

(32) A. Thum and W. Bautz, "Steigerung der Dauerhaltbarkeit von Formelementen durch Kaltverfornung," *Mitteilungen*, Technischen Hochschule, Darmstadt, No. 8 (1936).

(33) E. J. Warlow-Davies, "Fretting Corrosion and Fatigue Strength," *Proceedings*, Inst. Mechanical Engrs., Vol. 146, p. 32.

(34) E. E. Weibel, "The Correlation of Spring Wire Bending and Torsion Fatigue Tests," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 57, RP57-1, pp. 501–516 (1935).

DISCUSSION

Mr. H. F. Moore.[1]—I want to emphasize a statement which Mr. McAdam made, that it is hoped that there will be undertaken further investigations of the effect of combined stress. This "technical cohesive strength" is one of the important things just coming 'round the corner, and the effect of other kinds of structural damage, especially slip, upon it is of great importance.

Some years ago J. N. Kenyon, of Columbia University, showed that rotating a wire under bending, if a film of oil were forced between the wire and the jaws, did away with localized stress at the bearing to quite an extent. In recent experiments made in connection with bending, we have found that this localized stress could be at least mitigated by the use of a heavy coating of grease. Both of these cases, of course, would reduce the intensity of radial compression and would fall in line with what Mr. McAdam said.

Mr. M. F. Sayre.[2]—Mr. McAdam's paper has to be viewed in the light of several other papers which he has presented recently. Because of that, it is very difficult to discuss by itself.

I think the important thing which he has been bringing out is possibly this: the need of further study of other combinations of stress than the ones on which we have been concentrating our attention in the past.

We have had a series of theories of failure presented based on tests of a certain limited range of combinations. These theories of failure have dealt almost entirely with the conditions under which plastic yielding would begin. I think the evidence is becoming stronger and stronger that the major danger to structures comes not from plastic yield but from some form of brittle failure. This is evidenced by both published and unpublished information as to types of failure in welded and other structures and in machines. Consequently we will do well to emphasize our studies of cohesive strength rather than of plastic yield. I think it is also evident that no one simple theory of failure, such as the shear theory or the theory of maximum shear energy, and so on, will satisfy the picture, but that we will have to have some kind of three-dimensional diagram which will reflect the fact that sometimes it is a plastic yield failure and sometimes it is a cohesive failure, sometimes a fatigue failure, or a combination of these which must be considered in our studies of what will happen.

Mr. J. M. Lessells.[3]—I wish to associate myself with the remarks made by previous speakers on the importance of work on combined stresses.

Regarding the reference on page 15 to "chafing fatigue" the author seems to believe this is a manifestation of the influences of the combined radial compression and cyclic flexural stresses. There seems to be evidence[4] that

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[1] Professor of Engineering Materials, University of Illinois, Urbana, Ill.

[2] Professor of Applied Mechanics, Union College, Schenectady, N. Y.

[3] Associate Professor of Mechanical Engineering, Engineering Dept., Massachusetts Institute of Technology, Cambridge, Mass.

[4] L. E. Benson, discussion of paper by G. A. Tomlinson, P. L. Thorpe and H. J. Gough, "Fretting Corrosion of Closely Fitting Surfaces," *Proceedings*, Inst. Mechanical Engrs., Vol. 142, March, 1940, p. 401.

"chafing fatigue" will develop under movements which are normal to the contact surface. Flexural stresses do not appear to be necessary.

MR. D. J. MCADAM, JR.[5]—In regard to Mr. Lessells' statement about chafing fatigue, the paper does not intend to imply that fatigue failure cannot be influenced by the roughening of the surface or by the concentration of longitudinal stress due to press fits. The paper, however, calls attention to the possibility of failure under radial compression combined with a longitudinal stress range or even under repeated radial compression stress alone.

"Fretting corrosion" was studied very thoroughly by Tomlinson[6] and by Tomlinson, Thorpe, and Gough.[7] They showed that very slight repeated relative motion of two surfaces in contact will cause this type of erosion, even when the pressure is very slight, as in the mere contact of a sphere with a smooth steel plate. These investigators attribute the resultant erosion to the breaking of molecular cohesion. The effect of such erosion on the fatigue limit would depend, not on the pressure between the surfaces, but on the amount of roughening.

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[5] **Metallurgist, National Bureau of Standards, Washington, D. C.**

[6] G. A. Tomlinson, "The Rusting of Steel Surfaces on Contact," *Proceedings*, Royal Soc. (London), A, Vol. 115, pp. 472–483 (1927).

[7] G. A. Tomlinson, P. L. Thorpe and H. J. Gough, "An Investigation of the Fretting Corrosion of Closely Fitting Surfaces," *Proceedings*, Inst. Mechanical Engrs., Vol. 141, pp. 223–237 (1939). Discussion, pp. 238–244; also Vol. 142, pp. 401–406 (1939).

THE ABSOLUTE MEASUREMENT OF THE ENERGY LOSS IN A SINGLE STRIP OF SHEET IRON OR STEEL UNDER ALTERNATING MAGNETIZATION

By W. E. Shenk[1]

Synopsis

Consideration of the requirements which must be met if core loss is to be measured in absolute terms led to the development of an adequate method and of apparatus, which, after use over a period of years, has proved to be accurate, sensitive, yet rugged. Its advantage over the conventional Epstein test is that, besides yielding an absolute measure of core loss, it is non-destructive and eliminates the very appreciable effect of cold work because no shearing of the sample is required; yet the method can be adapted to a specimen of any size. This type of tester can thus be used not only for experimental investigations but also as a reliable means of grading electrical sheet.

Since the energy loss in sheet steel under alternating magnetization is one of the factors which limits the efficiency of certain electrical machinery, a basic requirement for the intelligent design of such equipment is a fairly precise knowledge of the magnetic quality of the steel to be used. Moreover, it is desirable that the method of measuring this loss be accurate, absolute, and non-destructive in the sense that it should be capable of testing a whole sheet of material, but none of the methods hitherto available has been satisfactory in all these respects. A method which meets these exacting requirements has been developed and has for some years been in successful use in production testing as well as in laboratory investigations. Its essential features have been given elsewhere[2]; the present paper contains a more detailed account, including a description of some of the special auxiliary instruments which had to be developed in order to make the method practicable.

The energy loss may be measured under either continuous (slowly varying) or alternating magnetization, the latter being usual in modern practice. The conditions required for a suitable measurement, and the types of apparatus best fitted for achieving them, are described by Lloyd and Fisher,[3] who state: "The specimen may be employed in three forms. (1) It may be in the form of straight strips placed in contact with a yoke, thus forming a closed circuit of ferromagnetic material. (2) The straight strips may be used without any yoke. (3) The specimen may be arranged to form a closed magnetic circuit in itself. The second form gives a distribution of flux which is far from uniform, and is therefore objectionable. The first form gives a more uniform flux, but it is necessary to distinguish between the energy supplied to the specimen and that supplied to the yoke. This can only be done satisfactorily by knowing the constants of the yoke, and only then by having the distribution of flux uniform, a condition difficult to secure. Consequently, for accurate measurements the third form is the most reliable, although for factory use the first or second may prove more convenient where accuracy can be sacrificed for other considerations.

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[1] Research Engineer, Research Laboratory, United States Steel Corp., Kearny, N. J.

[2] W. E. Shenk, U. S. Patent No. 2,176,279, October 17, 1939 (filed May 12, 1937).

[3] M. G. Lloyd and J. V. S. Fisher, "The Testing of Transformer Steel," *Bulletin*, Nat. Bureau Standards, Vol. 5, p. 453 (1908–1909).

"Assuming then a closed magnetic circuit of the material, to be tested by the wattmeter method, the following conditions should be realized as far as possible.

"1. The flux should be uniformly distributed over the cross-section of the specimen, and should be the same at every section. This requires that there should be no leakage of flux through the air.

"2. A definite form of wave of magnetic flux should be used, or in other words, a definite form of wave of secondary electromotive force, since the form factor of this wave enters into the computation of maximum flux density from the observed effective voltage.

"3. The material used should be cut in a form such that only a small part of it is contiguous to a cut edge, since it is well known that all methods of cutting have a hardening effect upon the material bordering upon the cut. This means that the strip, whether straight or in ring form, should not be too narrow. This condition may be dispensed with if all specimens are annealed under definite conditions after cutting to size, and prior to testing.

"4. The amount of material required should not be greater than is necessary to get a fair average value.

"5. The corrections to be made to the readings of the instruments should be small."

To these five conditions must be added a sixth, namely, that the magnetic measurements should not include material near a joint in the specimen. The presence of a joint disturbs the field in its vicinity and additional loss is introduced by eddy currents set up in the specimen by the component of the leakage flux normal to it. Schneider[4] has shown that in the Epstein apparatus this extra loss may amount to several per cent for a sample having a core loss of 2 watts per kilogram at a flux density of 10,000 gausses. With the improved steels now available, for which the core loss is only half as great, a joint has an even greater effect.

The earliest recorded attempt to devise an apparatus of this sort appears to be that of Richter,[5] who described a method in which a whole sheet is inserted in a toroidal coil, the power absorbed being measured by a wattmeter connected to the coil. This design is excellent magnetically, except for the joint formed where the ends of the sheet are brought together, but the magnetic properties of the material are so altered by the stress set up in bending the sheet that the results have little significance.

In 1929, Kinsley[6] designed a tester for whole sheets in which the magnetic circuit is completed through a laminated yoke. This method, though not absolute, can be calibrated against the Epstein test and is then useful in sorting and classifying sheets. Later, Dannatt[7] published a description of an apparatus for testing a single piece of sheet steel 12 in. long and up to 4 in. wide; it was not developed for use with a larger sample, although there is no reason why this cannot be done. The method yields an absolute value of the core loss characteristic of that portion of the sample in which the flux is not disturbed by leakage, but the power must be calculated from the readings of an a-c. potentiometer which is a time-consuming procedure. Gunderson[8] has developed an apparatus, based upon a principle substantially identical with that used by Kinsley, which in several respects resembles Dannatt's apparatus; at least part of the yoke loss is included in the wattmeter reading, however, so that this apparatus must be calibrated against some standard test. Another single-strip tester, limited to the testing of a strip 10 in. long and $1\frac{3}{16}$ in. wide, has been devised by Smith[9]; this tester measures the yoke loss as well as the loss in the specimen and must therefore be calibrated against some other method of test. Smith recommends calibration against the Epstein test, but in view of the small size of the specimen relative to the standard Epstein sample (the ratio is about 1/550) this comparison is extremely tedious and the validity of such a calibration is open to question. The method is, however, useful in determining a difference between samples and in experimental work, since it is possible to measure the same small sample before and after a given treatment.

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[4] J. Schneider, "Die Messung der magnetischen Verluste an Eisenblechenstreifen bei homogener Sättigung," *Archiv für Elektrotechnik*, Vol. 24, p. 651 (1930).

[5] R. Richter, "Apparatus for Testing the Electromagnetic Quality of Large Sheets of Iron," *Elektrotechnische Zeitschrift*, Vol. 23, p. 491 (1902); Vol. 24, p. 341 (1903).

[6] C. Kinsley, U. S. Patent No. 2,101,780, December 7, 1937 (filed October 8, 1932).

PRINCIPLE OF THE METHOD

The theory underlying the measurement of the magnetic properties of a portion of a ferromagnetic circuit is largely due to Rogowski.[10] The energy loss in an iron core under cyclic magnetization may be regarded as the integral of the magnetic potential and the flux density taken over a cycle and over whatever volume of the core is being considered, a definition which is perfectly general and is not limited to homogeneous magnetization of the core and the adjacent air space. Usually, however, we deal with the restricted case of a uniform field and regard the core as uniform in properties, at least over the element of volume. In general, however, the core is not uniform so that, when we consider it to be so, we are in fact considering the average energy loss over the volume of core included in the measurement.

The energy loss per cycle per unit volume in a homogeneous iron core of uniform cross-section, in which the magnetic field is uniform and parallel to the axis of the core, is:

$$\text{Energy loss} = \frac{1}{4\pi}\int H\,dB\ldots(1)$$

and

$$\text{Power loss} = \frac{f}{4\pi}\int H\,dB\ldots(2)$$

where H = instantaneous magnetizing force in oersteds,
B = instantaneous flux density in gausses, and
f = frequency in cycles per second.

If the field is produced by a uniformly-wound solenoid of infinite length, and the portion of the core under consideration is regarded as part of an infinitely long core of uniform cross-section, the field H, that is, the magnetic potential gradient, is parallel to the axis of core and solenoid and has the value

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[7] C. Dannatt, "Energy Loss Testing of Magnetic Materials Utilizing a Single Strip Specimen," *Journal of Scientific Instruments*, Vol. 8, p. 276 (1933).

[8] D. T. Gunderson, U. S. Patent No. 2,138,965, December 6, 1938 (filed August 17, 1933).

[9] T. G. Smith, "A Tester for Measuring Core Loss of Single Strips," *General Electric Review*, Vol. 40, p. 273 (1937).

[10] W. Rogowski, "Die Messung des elektromagnetischen Energieflusses," *Archiv für Elektrotechnik*, Vol. 4, p. 279 (1916).

$$H = 4\pi n i \text{..........} (3)$$

where n = turns per centimeter of length of the solenoid, and

i = magnetizing current flowing in the solenoid winding.

If a uniform secondary coil is wound on the portion of the core whose properties are being determined, an electromotive force e will be set up in it such that

$$e = Na \frac{dB}{dt} \text{.........} (4)$$

where N = total number of turns in the secondary winding, and

a = cross-section of the core in square centimeters.

The value of dB derived from Eq. 4 and that of H derived from Eq. 3 may then be substituted in Eq. 2 to give the power per unit volume of the core which is

$$\text{Power} = \frac{fn}{Na} \int ei\,dt \text{......} (5)$$

Now, if a wattmeter is connected in series with the solenoid, so that its current coil carries i abamperes, and there is an electromotive force e impressed across its potential coil, this instrument indicates the time average ei product,

$$f \int e\,i\,dt \text{..........} (6)$$

that is, the integral in Eq. 5 may be evaluated for the circuit in question by means of a wattmeter. The core loss in absolute units per cubic centimeter of iron is then

$$\text{Core loss} = \frac{nW}{Na} \text{ abwatts}^{*} \text{ per cubic centimeter..} (7)$$

where W = wattmeter reading in abwatts.

Converting Eq. 7 to practical units and expressing the loss in terms of unit weight of iron instead of unit volume, we have

Core loss

$$= \frac{1000nW'}{Na\rho} \text{ watts per kilogram, or}$$

$$P_{B/f} = \frac{453.6nW'}{Na\rho} \text{ watts per pound} \quad (7a)$$

where $P_{B/f}$ = core loss in watts per pound at maximum flux density B and frequency f,

ρ = density of the iron in grams per cubic centimeter, and

W' = wattmeter reading in watts.

It has been assumed thus far that the loss in the wattmeter potential circuit is substantially zero. If this is not the case, this additional loss must be subtracted from the reading of the instrument to get W', but as is shown later, this loss may be automatically excluded from the wattmeter reading.

It is thus demonstrated that a wattmeter can be used to evaluate the power integral Eq. 2, providing Eqs. 3 and 4 are satisfied, the more significant being Eq. 3 which holds only for a perfectly uniform field such as that inside an infinite solenoid. It is not necessary, however, to have an infinite solenoid to produce such a field, for a short solenoid suffices *if there is compensation for the radial component of the field at its ends.* The criterion for the exact amount of this end compensation is provided by Eq. 3. Thus, it is necessary only to

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[*] The abwatt is the absolute unit of power and is equal to 10^{-7} watts; an abampere is 10 amp., and an abvolt is 10^{-8} volts.

measure H and i in some manner and adjust the end compensation until Eq. 3 is satisfied. This end compensation can be achieved in principle by adding an infinitely-long, uniformly-wound solenoid to each end and connecting these in series with each other and with the short solenoid and extending the core to infinity, thus fulfilling the criterion by definition. The other alternative is to energize these infinite end coils from a separate source, and to adjust the current until Eq. 3 is satisfied for the main coil. Obviously, if conditions required by Eq. 3 can be satisfied by coils and cores added to the ends of the short solenoid, it is unnecessary to extend the added ends to infinity; an iron yoke of any convenient size may be used to complete the magnetic circuit and the end magnetization or compensation can then be supplied from windings on this yoke.

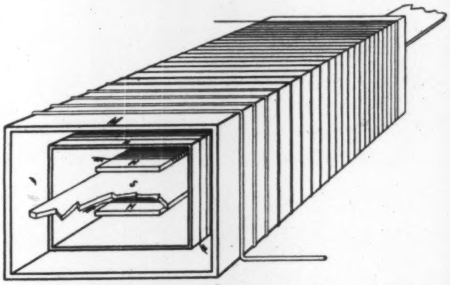

FIG. 1.—Schematic Diagram to Show Relative Positions of Magnetizing Winding M, B Winding, H Coils, and Specimen S.

Some means of measuring H in terms of i is required to carry out this compensation. The most direct way of determining H is to measure the axial component of the air field within the magnetizing coil near the core by means of a small flat exploring coil with merely an air core; this coil is wound on a card of insulating material and its turn-area is accurately known. The ratio of H to i, which is more significant than the actual value of H, may be measured with the aid of an air-core mutual inductance whose primary is connected in series with the magnetizing winding and whose secondary is connected in series opposition to the H coil. An alternating current galvanometer in the secondary circuit enables the output of the H coil to be compared with that of the mutual inductance, and the yoke magnetization may then be adjusted until the desired ratio of H to i is obtained. The mutual inductance is adjusted to have the value

$$4\,\pi\, n N_{\mathrm{H}} A \ldots\ldots\ldots\ldots (8)$$

where A = cross-section area of the H coil in square centimeters, and

N_{H} = total number of turns in the H coil.

The ratio of H to i is then correct when the galvanometer indicates zero, thus indicating the correct value for the yoke magnetization.

If the H coil is placed next to the iron core (specimen) as shown schematically in Fig. 1, and the secondary which is used for the potential circuit of the wattmeter is wound around the H coil as well as the iron, the correct ratio of H to i is established inside of the potential secondary or B coil. Consequently, the losses in the potential circuit of the wattmeter are not included in the power loss reading, since the conditions required by the theory are established at the iron. It follows that any other load connected to this secondary, or to any similar secondary, will not affect the wattmeter indication so that another instrument such as a voltmeter may also be connected to the B coil.

The energy loss in this iron must be measured at a known maximum flux density $B_{\mathrm{max.}}$ if it is to conform to accepted practice. The flux density

$B_{\text{max.}}$ may be determined from the reading of a voltmeter connected to the B coil; preferably this should be an average voltmeter, that is, an ordinary d-c. voltmeter or potentiometer connected to a precision mechanical rectifier. Such an average voltmeter indicates the average electromotive force per half cycle induced in the B coil; this emf. is uniquely related to the maximum flux linking the coil, that is, it is independent of the wave form of the time variation of the flux. This relation between the average voltage per half cycle $E_{\text{avg.}}$ and the maximum flux density $B_{\text{max.}}$ is

$$E_{\text{avg.}} = 4faNB_{\text{max.}} \text{ abvolts}$$

or

$$E_{\text{avg.}} = 4faNB_{\text{max.}} \times 10^{-8} \text{ volts} \ldots (9)$$

Besides making core loss measurements, this method can be used to obtain the magnetization curve and permeability under alternating magnetization as will be described in a future paper.

This is the complete theory of the single strip tester. The actual apparatus incorporates certain refinements which will be discussed in the detailed description of the apparatus.

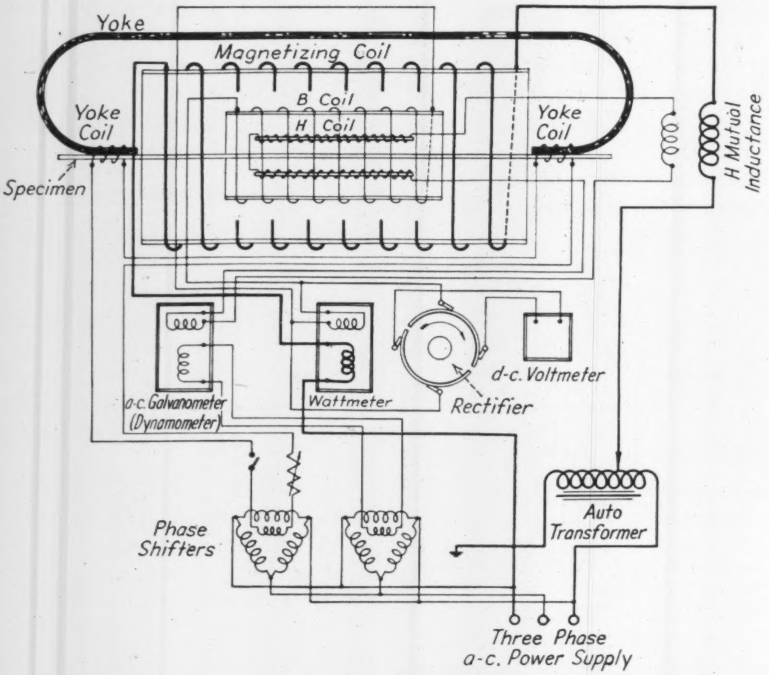
Yoke
Magnetizing Coil
B Coil
H Coil
Yoke Coil
Yoke Coil
Specimen
H Mutual Inductance
a-c. Galvanometer (Dynamometer)
Wattmeter
Rectifier
d-c. Voltmeter
Auto Transformer
Phase Shifters
Three Phase a-c. Power Supply

FIG. 2.—Simplified Circuit Diagram Showing Essential Elements of Apparatus; Magnetizing Circuit Indicated by Heavy Line.

APPARATUS

The theory leads to an apparatus whose essentials are shown by the simplified diagram in Fig. 2. The wattmeter is connected to the magnetizing winding that acts directly upon the specimen and to the secondary that is wound around the specimen. A voltmeter and commutator are connected together and to the secondary so that the maximum flux in the specimen can be measured correctly regardless of the time-wave form of the flux. The mutual inductance for the measurement of the ratio of H to i is shown with its primary connected in series with the main magnetizing coil and its secondary connected in series opposition to the H coil. The correct ratio of H to i is established by adjusting the current supplied to the yoke magnetizing coils, in phase as well as in amplitude, until the a-c. galvanometer (dynamometer) connected in this circuit does not deflect. In thus arriving at the correct loss in the specimen to be tested, no other type of magnetic test has been used; the energy loss in the specimen is determined entirely from the coil constants of the apparatus and the wattmeter reading, so that this method yields an absolute result. The apparatus resembles superficially that of Kinsley,[6] Gunderson,[8] or of Smith,[9] but differs significantly from these in that the method is absolute; it requires no calibration, being independent of the properties of the specimen being tested and of the iron in the yoke, and depends only upon the indication of standardized instruments.

This type of tester has been built and operated in four different sizes, the maximum width of specimen being 3, 9, 27 and 36 in., respectively. The smallest is convenient for laboratory work, whereas the largest is designed for production testing of whole sheets. All sizes are, however, used in production testing of steel produced as a continuous strip.

In order to satisfy the requirements discussed earlier, the apparatus must comprise a uniformly-wound magnetizing coil, a secondary, a yoke with windings upon it, an H coil whose constants are known, a wattmeter for the power measurement, a voltmeter to be used in determining the magnetic flux, and certain auxiliary apparatus which is described later. The main magnetizing coil must be very uniformly wound and its length must be accurately known in order that the core loss may be determined from the instrument readings. The number of turns in the secondary coil must be accurately known but the coil dimensions are not significant so long as they remain fixed. The H coil must be permanent in dimensions and must have a sufficient turn-area to enable the air field to be measured to the required accuracy. The yoke need only be of large cross-section relative to the

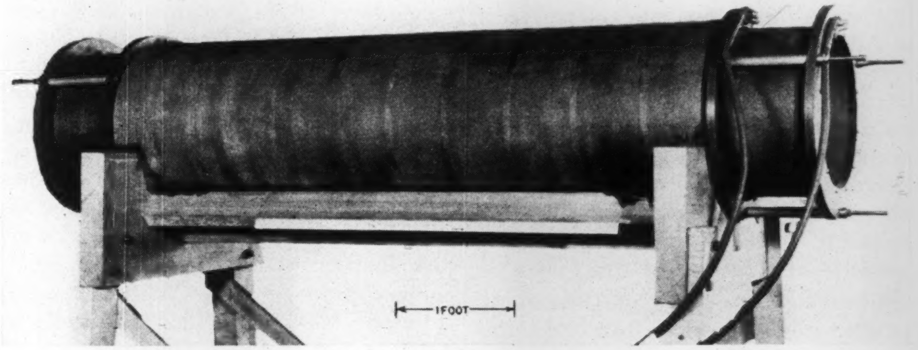

FIG. 3.—Standard Solenoid (7 ft. Long Over-All, 14 in. Minimum Diameter) to Set up a Uniform Absolute Magnetic Field.

cross-section of the largest specimen to be tested and must be made of good ferromagnetic material; the only requirement is that the flux density corresponding to the maximum permeability of the yoke iron shall not be exceeded at the highest flux required for the largest specimen to be tested.

Apparatus for Measuring Ratio of H to i:

As it is inaccurate as well as inconvenient to measure the *H* coil voltage and the magnetizing current in order to determine whether the proper voltage-current ratio for a perfectly uniform field within the magnetizing coil has been established, an air-core mutual inductance is used as an aid to the accurate determination of this ratio. An air-core mutual inductance has a fixed ratio of secondary voltage to primary current determined by the constants of the inductance, hence, such a mutual inductance with the proper constants provides a criterion of the voltage-current ratio that must be maintained between the *H* coil voltage and the magnetizing current to insure a perfectly uniform field. The proper voltage-current ratio could be calculated from the turn area of the *H* coil and the turns per unit of length in the magnetizing coil, and the air-core mutual inductance would then be constructed to have this ratio; but the *H* coil has very small dimensions so that its turn area cannot be accurately calculated and must therefore be determined by measurement of the voltage induced in the coil when it is placed in a perfectly uniform alternating magnetic field. It is necessary therefore to construct a solenoid large enough to produce a perfectly uniform magnetic field over a volume sufficient to permit the *H* coils in the largest tester to be placed entirely within the region of uniformity. Such a solenoid, shown in Fig. 3, follows the design of Bestelmeyer[11] but is more than twice as large; in fact, the calibration of the *H* coils for the largest tester required the construction of a solenoid capable of producing a uniform magnetic field of larger volume than any other of which we are aware. The coil consists of three sections; a main section about 5 ft. in length and 16 in. in diameter, and two end sections each about 1 ft. in length and 14 in. in diameter. The cylinders upon which these coils are wound are threaded with a round bottom groove of exactly seven turns per inch and the single layer of No. 8 double-cotton covered magnet wire is wound in this helical groove. This method of winding assures uniform turn spacing over the whole length of each of the three coils. From the dimensions, the turns per unit of length of each of the three coils, and the position of the auxiliary coils relative to the main coil, the field along the axis of the solenoid can be calculated by well-known methods. The end coils can be adjusted relative to the main coil to produce the optimum conditions as to uniformity of the magnetic fields along the coil axis. As a result of the accuracy with which all dimensions are known, the field at the mid-axis of the main coil is known to be within 0.01 per cent of the value it would have in an endless solenoid. By actual measurement the field is uniform within less than 0.05 per cent with respect to the value of the center at all points within the middle 2 ft. of the main coil; the departure from uniformity is about 0.02 per cent over the volume actually used in the calibration of the *H* coils.

Each *H* coil, which consists of a single layer winding of fine copper magnet wire wound on a flat card of Bakelite, was cemented with Bakelite varnish and baked between clamps for 24 hr. at 250 F., a procedure which insures that the turn area of the coil will remain sufficiently constant. In each single strip tester several of these *H* coils are used rather than a single large coil because the turn area of a large thin flat coil would not be constant. The *H* coils were assembled on the thin Bakelite liner, as shown in Fig. 4 for one of the 3-in. testers; they were then covered with two layers of thin cotton cloth, varnished with Bakelite, covered with two thin sheets of copper (one on each side of the flat coil) to shield against capacity coupling; then with more cloth and varnish and finally the secondary or *B* coil was wound directly over the *H* coil. The final assembly, shown in Fig.

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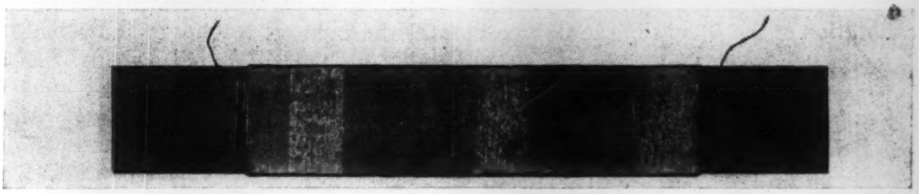
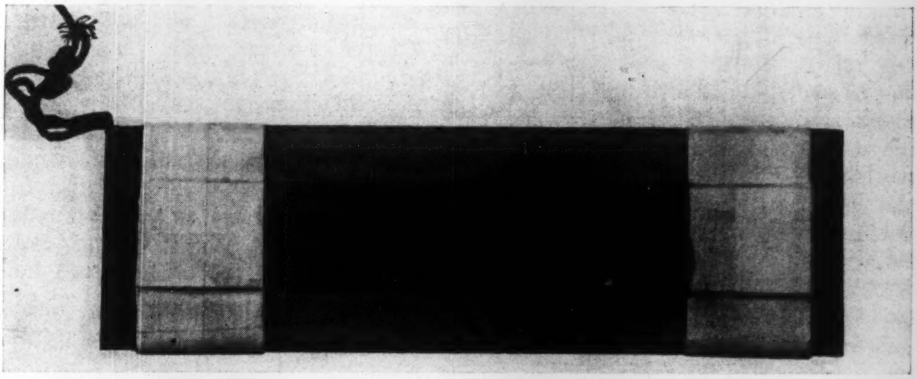
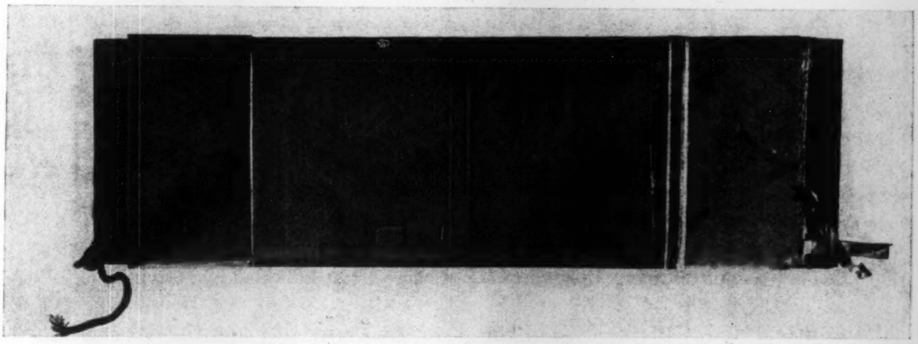
[11] A. Bestelmeyer, "Berechnung, Herstellung und Messung eines homogen Magnetfeldes," *Physikalische Zeitschrift*, Vol. 12, p. 1107 (1911).



(*a*) An *H* coil.



(*b*) Pair of *H* coils assembled on hollow insulating form.



(*c*) *B* coil wound over *H* coil assembly (*b*).

FIG. 4.—Steps in the Assembly of the *B* and *H* Coils of the 3-in. Single Strip Tester.

4 was baked at 250 F., after which it became essentially a solid and very rigid tube of Bakelite and cloth with copper windings in the walls.

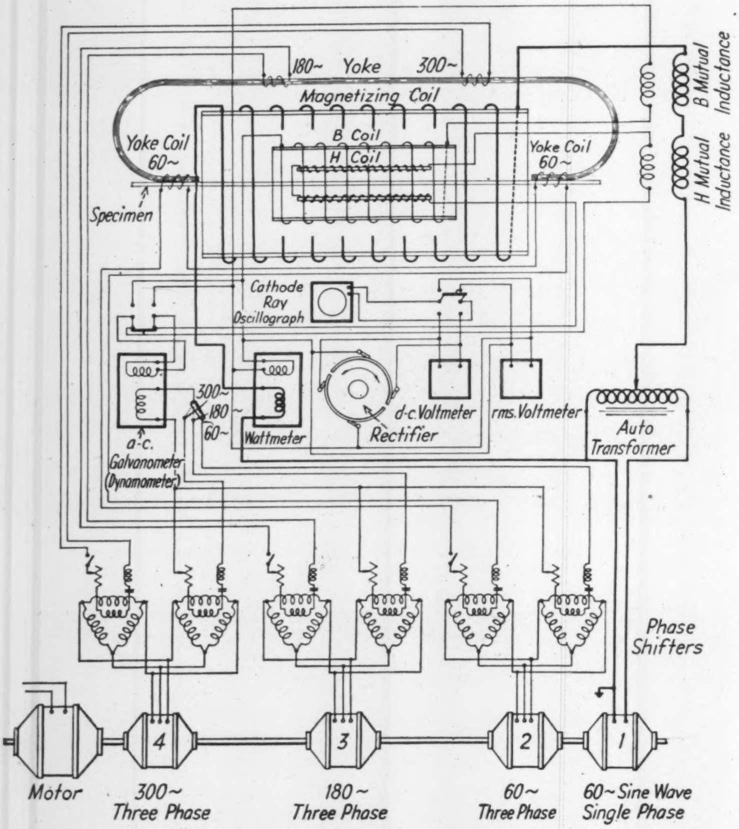

180~ Yoke 300~
Magnetizing Coil
B Coil
H Coil
Yoke Coil 60~
Yoke Coil 60~
Specimen
B Mutual Inductance
H Mutual Inductance
Cathode Ray Oscillograph
a-c. Galvanometer (Dynamometer)
300~
180~
60~
Wattmeter
d-c. Voltmeter
Rectifier
rms. Voltmeter
Auto Transformer
Phase Shifters
4
3
2
1
Motor
300~ Three Phase
180~ Three Phase
60~ Three Phase
60~ Sine Wave Single Phase

FIG. 5.—Complete Circuit Diagram; Magnetizing Circuit Indicated by Heavy Line.

The 3 and 9-in. tester coils were completely assembled in this manner before calibration of the *H* coils in the standard field was attempted. In the case of the 27 and 36-in. tester only the *H* coils were assembled on a card of Bakelite 12 in. wide by 28 in. long for calibration, since the full coil assembly was too large for insertion in the standard field. After the assemblies of the coils had been made in this manner the absolute calibration of the *H* coils in the standard field was made. As a first step, the *H* coil assembly was placed in the standard field in an axial position, the standard solenoid being energized with about 10 amp. of 60-cycle alternating current, and the induced voltage in the *H* coils was measured. From the data obtained in this measurement the proper dimensions for the air-cored mutual inductance required for the accurate measurement of the voltage-current ratio mentioned previously could be calculated. The mutual inductance, so constructed that a final fine adjustment in its constants of

± 1 per cent could be made, was made in accordance with these calculations and was adjusted to within 1 per cent of the desired value. For the final adjustment this mutual inductance must be compared with the *H* coils which were placed in the standard solenoid; to this end the primary of the mutual inductance and the standard solenoid are temporarily connected in series so that the same current flows through each. The secondary voltage of the mutual inductance is compared with that of the *H* coils by means of a Maxwell bridge circuit and the final adjustment of the inductance is made to the limiting accuracy of the bridge used, about 0.02 per cent. With the completion of this adjustment the complete tester is calibrated absolutely in terms of the dimensions and turns per unit length of the standard solenoid and of the turns per unit length of the magnetizing coil of the tester.

Elimination of Electromotive Force Induced by Air Flux:

There remains one small correction to be applied to the tester to eliminate the emf. induced in the *B* coil by the alternating air flux that links it. This emf. adds to that due to the ferric induction and introduces an error which can be eliminated by means of an air-core mutual inductance which compensates for the undesired emf. as in the case of the measurement of *H*. This provides the proper correction because the air field linking the *B* coil is numerically equal to *H* and in fact is the same field that is measured to determine *H*. The proper value for the mutual inductance can be determined with the aid of the standard solenoid used in adjusting the mutual inductance for the *H* coils, the procedure being exactly the same as in the case of the *H* coils. The secondary coil assembly of the tester is placed in the standard field, the induced voltage in it is measured for a particular value of the field, and the proper size of the mutual inductance is then calculated from these data. As in the case of the *H* coils the final adjustment of the mutual inductance is made by comparison of the mutual inductance with the *B* coil.

Arrangement of Circuit:

In Fig. 5 the schematic circuit arrangement of the original strip tester is shown and the tester itself is shown in Fig. 6. The present testers differ from this only in minor details. (The 36-in. tester is shown in Fig. 7.) The magnetizing coil is shown extending between the ends of yoke and the secondary windings are shown inside of the magnetizing coil. There are four main circuits which must be considered, namely, the primary or magnetizing circuit, the secondary or *B* circuit, the *H*-measuring circuit, and the yoke-magnetizing circuit. The magnetizing circuit is shown supplied from a 60-cycle alternating-current generator No. 1 (Fig. 5) through the auto-transformer. This circuit consists of the primary of the two mutual inductances connected in series with the magnetizing coil and the current coil of the wattmeter. The secondary, or *B* circuit, consists of the *B* coil and the secondary of one of the mutual inductances connected to the potential coil of the wattmeter, a commutator, and two voltmeters. The *H* measuring circuit consists of the *H* coils connected in series opposition to the secondary of the other mutual inductance and to one element of an a-c. galvanometer or dynamometer. This instrument is used to detect the presence of any current flowing in the *H* circuit. The fourth circuit, the yoke magnetizing circuit, consists of coils

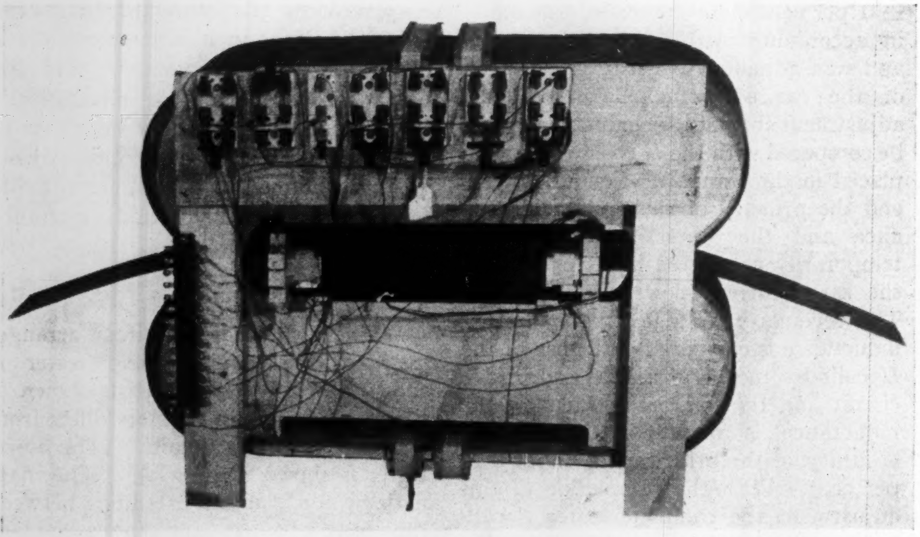


FIG. 6.—Original Single Strip Tester, Showing Double Yoke Initially Used, and Pairs of Yoke Magnetizing Coils.

FIG. 7.—Large (36 in. Wide) Single Strip Tester as Installed in a Mill.

on the yoke which are supplied from alternating-current generators No. 2, 3, and 4 through phase-shifting transformers and rheostats so that the yoke magnetization may be adjusted in both phase and magnitude.

Measurement:

Before proceeding to a discussion of the actual measurement of core loss, certain precautions that must be taken in the circuits for measuring H and B must be considered. Due to the high sensitivity required in the measurement of H, it is important to eliminate all electrical leakage into the circuit which includes the H coils, the secondary of the air-core mutual inductance that is used as a criterion of the correct ratio of H to i, and the null dynamometer moving coils. By carefully insulating and twisting all connections in this circuit, conductive and inductive leaks, respectively, are readily eliminated, but this is not sufficient. Due to the minute emf. developed in the H coils, the small displacement currents flowing through capacities between the leads and coils on the one hand and external circuits on the other hand, which in most cases would be unimportant, can seriously affect the deflection of the null dynamometer. An effective way[5] of reducing this capacity current is to enclose the twisted leads of this circuit in a grounded metallic sheath, shield between the B and H coils as mentioned previously, shield between the moving coils of the dynamometer and the field coils, and ground these shields, the yoke, and the side of the magnetizing circuit connected to the current coil of the wattmeter all to the same ground connection. It is only slightly less important to eliminate similar electrical leaks from the potential circuit of the wattmeter, which can be accomplished in a similar manner. It would be expected that these leaks would be especially important in the smallest tester (3 in.), but as shown by suitable crucial tests their complete elimination demonstrated the full effectiveness of these precautions.

In measuring the core loss in the specimen, the cross-sectional area of the specimen is measured before starting the test and the proper B-coil voltage for the desired maximum flux density (usually $B_{max.} = 10{,}000$ gausses) is calculated for this cross-section. This calculation is made according to the formula

$$V_{avg.} = (4fNaB_{max.}) \times 10^{-8}$$

where $V_{avg.}$ = average voltage per half cycle,
$B_{max.}$ = flux density in gausses in the specimen,
N = number of turns in B coil,
a = cross-section area of the specimen in square centimeters, and
f = frequency in cycles per second.

The specimen is now inserted in the tester and the magnetizing current adjusted until the d-c. voltmeter indicates the calculated value of $V_{avg.}$ when the flux density desired is attained in the specimen. It will be noted that the alternating current galvanometer is deflected indicating that the desired uniform field conditions are not present in the magnetizing coil. The next step is to energize the yoke windings from generator No. 2 which supplies 60-cycle alternating current and so adjust this current in magnitude and phase that the galvanometer deflection becomes zero. The wattmeter will then indicate the correct value of the core loss because the yoke magnetization has now been adjusted so that the perfectly uniform field conditions of an infinite solenoid have been established. The power loss in the potential circuits of the wattmeter and voltmeter does not appear in the wattmeter reading because the *H* coil is within the *B* secondary.

There is one more aspect of this test that must be considered. At the beginning of this paper certain conditions for an ideal magnetic test under alternating magnetization were stated, one of which was that the wave form of the alternating magnetic flux be sinusoidal or that the wave form should be measured. It is very important to know the wave form exactly because core loss is a function of wave form. The wave form of the secondary voltage of the tester can be determined from the readings of two voltmeters, one of which is an ordinary alternating current instrument which measures the a-c. or effective value of the voltage; the other is a direct-current voltmeter which is connected to a mechanical rectifier and measures the average value of the rectified secondary voltage. The ratio of the effective voltage to the average voltage is called the form factor and has the value 1.11 for a perfect sine wave. If during the test the ratio of these two voltmeter readings differs by more than 1 or 2 per cent from the sine wave value it is necessary to apply additional magnetization of odd multiples of the frequency of the testing supply, either to the yoke or to the main magnetizing circuit. In Fig. 5 the additional third harmonic magnetization is applied to the yoke by means of separate windings on it from generator No. 3 and provision is also made to supply the next higher odd harmonic magnetization through other yoke coils from generator No. 4. In our final apparatus we have incorporated only the third harmonic correction and it has been found better, especially in the large testers, to apply this additional harmonic magnetization in the magnetizing circuit, but the principle of wave form correction is the same in either case. We can use the dynamometer used in determining when the *H* difference becomes zero to measure the amount of third harmonic voltage across the terminals of the *B* coil. A dynamometer has the useful property of measuring only current of the same frequency as is used to excite its field coils, hence, if the field coils of the dynamometer are energized with third harmonic current (180 cycles) the instrument ignores the fundamental (60 cycle) component of the *B*-coil voltage and measures the harmonic component. The extra magnetization of 180-cycle frequency applied to the tester is then adjusted both in phase and amplitude until the dynamometer deflection is reduced to zero.

By this means, the 180-cycle component is eliminated from the wave form of the flux in the specimen and since this harmonic is the principal distortion introduced in this type of a test, the flux wave form is then essentially sinusoidal. The ratio of the voltmeter readings indicates whether the form factor is now close enough to the sinusoidal value for our purposes. If this is the case and the flux density in the specimen and the yoke magnetization are correct, the wattmeter will indicate the true core loss in the specimen under the specified conditions. If, however, the form factor is still more than 2 per cent from the value 1.11, a correction must be made in the core loss value; for example, in accordance with the method outlined in the paper by Smith and Concordia.[12]

Wattmeter and a-c. Galvanometer:

The power to be measured in the 3-in. tester is of the order of 0.04 watt for testing at 10,000 gausses maximum flux density and, since it is desired that the over-all accuracy of the core loss measurement be within ± 1 per cent, the watt-meter must be capable of measuring 0.04 watt to better than 0.4 of a milliwatt. Furthermore, the current coil resistance in this instrument must be extremely low (not over 0.01 ohm) and the power consumption in the potential circuit should be held to a small part of the total power being measured. These requirements make it necessary to resort to a suspended moving coil type of wattmeter, but at the time this development was started, no suitable instrument was available, hence, it was necessary to design and build one. One of the most difficult problems in connection with this instrument was to find a suitable material for the suspension, which must have a low enough torque so that unnecessarily heavy moving coils do not have to be used and at the same time must have an elastic aftereffect as nearly zero as possible. We were able to find only two materials that satisfied this specification: namely, fused quartz and tungsten. Since the suspension forms one of the connections to the moving coils and must therefore be a conductor of electricity, fused quartz is eliminated.[13] Tungsten, which is only

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[12] B. M. Smith and C. Concordia, "Core Loss Measurements at High Flux Densities," *Transactions*, Am. Inst. Electrical Engrs. (1931).

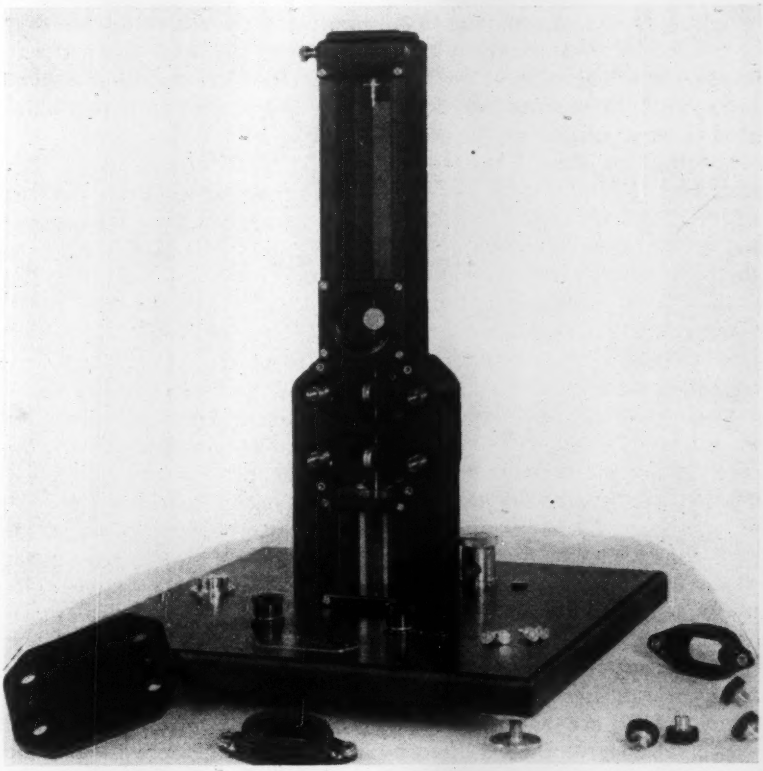

FIG. 8.—High-Sensitivity Wattmeter (Dynamometer).

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[13] Metallized fused quartz filaments are not satisfactory because of the short life of the conducting metal film.

slightly inferior to fused quartz in elastic after-effect and is a sufficiently good conductor of electricity, is therefore about the only material satisfactory for this purpose.

The instrument was designed to be as nearly astatic as possible so that the presence in its vicinity of an alternating field would have no significant effect upon its accuracy. A micrometer for adjusting the zero of the instrument was built into the top of the support for the suspension so that the moving coil could be adjusted to zero position on the scale without opening the case. The completed instrument, built for us by the Weston Electrical Instrument Co. after more than a year of investigation in our Laboratory followed by 2 yr. development on their part, is shown in Fig. 8. All the material that went into this instrument was carefully selected so as to be free from magnetic contamination and the final assembly was made in a dust-free, air-conditioned room to further insure against the presence of such contamination, particularly in the moving coils.

Figure 8 shows the instrument with the front cover removed to expose the moving coils. Two of the four current coils or field coils, which are removable, have been taken out and are lying at the base of the instrument. Three sets of these current coils are supplied with each instrument to give three different current ranges: namely, $\frac{1}{4}$ amp., 5 amp., and 25 amp. This flexible combination makes it possible to obtain a full scale deflection over the million fold range 1 milliwatt to 1 kw.

The a-c. galvanometer in the tester is exactly like the wattmeter in appearance and differs in design only in minor details, but it presented a much more difficult problem to the instrument maker. This instrument must have a current sensitivity comparable to the best d-c. laboratory galvanometer, and yet must be to a very high degree insensitive to disturbing fields and mechanical vibration; moreover, the deflection and the zero position must be stable to a much higher degree than is actually attained in most d-c. galvanometers of comparable sensitivity. The sensitivity of this instrument is 4×10^{-10} amp.^{2} per mm. with the scale at 1 meter distance and since the field coil current is 0.1 amp. the corresponding current sensitivity is 4×10^{-9} amp. per mm.

The Voltmeters:

The voltmeters were not nearly so difficult to obtain as the dynamometers. They are of standard design, but, since the power consumed by them must be a small fraction of the total power being measured, they had to be especially built to suit the tester; furthermore, the range of specimen size and flux density to be covered by each tester necessitated about twenty different ranges on the instrument. This could not be obtained in one instrument except by the use of external multipliers so the multipliers and the range switch had to be mounted separately. The Weston Electrical Instrument Co. was most helpful in solving this problem.

The Commutator:

Since the only completely satisfactory way to measure the maximum flux density in an alternating field is by rectifying the voltage in a coil linking the field and measuring the d. c. or average value of this rectified voltage, some sort of rectifier must be employed. It was very soon decided that vacuum tube and other electronic rectifying devices were unsuitable, since Dannatt and Holt[14] have shown that at voltages below 100 volts the error introduced is too great. At 20 volts (the highest reached in any of the testers) the error is nearly 7 per cent for even the best of these devices. This indicated the desirability of using a mechanical commutator of the type described by Dannatt and Holt, but the construction of a suitable one was no simple task. The basic difficulty is to get the brushes to stay on the commutator. After making repeated changes in the design of the brush holder we were almost convinced that all brushes jump and stick. The difficulty was finally overcome, however, by the use of the Baylis reaction brush holder,[15] which is just a plane surface inclined at an angle of about 60 deg. to the commutator in its plane of rotation. The brush is cut in a special shape and held against the holder and the commutator by spring pressure. The friction between the brush and the rotating commutator reduces the brush pressure against the holder to nearly zero so there is practically no friction between the brush and holder. The reaction type brush holders used for the rotor brushes are shown in Fig. 9.

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[14] C. Dannatt and N. Holt, "A Precision Average Voltmeter for Power Frequencies," *World Power*, Vol. 7, p. 241 (1927).

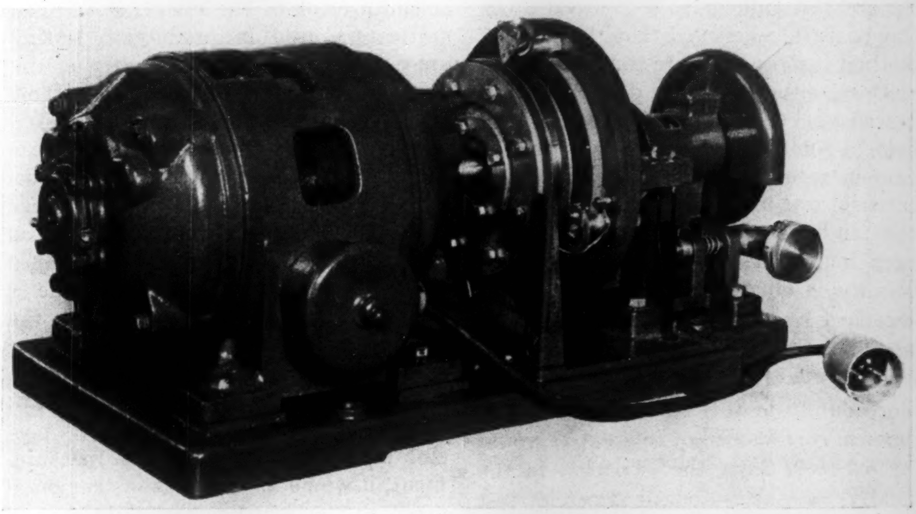

FIG. 9.—Precision Commutator (Rectifier).

With this problem solved, the design was modified to incorporate other improvements which seemed desirable in the light of accumulated experience; the final model, of which four were built to our design by the Automatic Temperature Control Co. of Philadelphia is shown in Fig. 9. Its main characteristics are rigidity, accurate alignment of commutator and brush rigging, perfect balance of rotating parts, and a suitable means to rotate the brush rigging so that rectification will occur at the zero of the alternating voltage. An indication that proper rectification is achieved is provided by an oscillograph permanently connected in the circuit.

The thermal electromotive force set up at the brushes is negligible so that an emf. of as low as 10 mv. can be rectified with an error of less than 0.1 per cent;

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[15] The Baylis brush holder is manufactured by the Baylis Co., Bloomfield, N. J.

in fact, this commutator has been used in connection with a galvanometer and type *K* potentiometer for measuring a-c. voltages as low as this with the accuracy limited by the potentiometer and not the commutator.

ABSOLUTE ACCURACY OF THE APPARATUS

The accuracy of any method of magnetic testing depends in principle upon (*a*) the degree of uniformity of the field in the specimen, (*b*) the accuracy of measurement of the properties desired in that portion of the specimen in which the greatest uniformity is attained. On this basis it is believed that the present method is more accurate than any other method, either a. c. or d. c., hitherto described. Careful measurements of the field in some of the preliminary models demonstrated that at all flux densities, the field was uniform within ± 0.03 per cent; in later models, the uniformity has been improved. Such a degree of uniformity is not even approached in the standard Epstein test using butt joints, for which a deviation of about 12 per cent at $B_{\text{max.}} = 10{,}000$ has been observed. Fortunately in this test the percentage error in core loss is not so great as would be expected from the variation in the field, else the standard Epstein test would be seriously in error by an amount depending upon the magnetic quality of the sample. This is in part due to other errors in the Epstein test which, under certain circumstances, tend to compensate partially for the nonuniform distribution of flux.

Condition (*b*) above is essentially a consideration of the instrumental errors involved in the measurement. In the present method the instruments whose accuracy influences the result are:

1. An instrument for measuring voltage. For laboratory work this is preferably a potentiometer of the Leeds and Northrup K2 type; for production testing a calibrated voltmeter is used.
2. A wattmeter.
3. A reflecting dynamometer of high sensitivity and high stability as regards the mechanical zero.

Since the flux density in the sample is adjusted to be correct with the aid of the voltmeter or potentiometer and since the core loss is in error by nearly twice the error in setting the flux density, the instrumental error introduced in the absolute accuracy of the method by these instruments is about twice the absolute error of the instrument. The voltmeters used in production testing are accurate to $\frac{1}{4}$ per cent, hence, the error introduced is about $\frac{1}{2}$ per cent. The accuracy of the voltage measured by a potentiometer can be made so great that its error is entirely negligible compared to other errors. On the other hand, there may be an uncertainty as great as ± 0.10 per cent in the resistance of the circuits in which the potentiometer is used, hence, the over-all error in the voltage measurement may be as great as ± 0.10 per cent.

The wattmeter used is accurate to within 0.1 per cent. Adding this possible error to that in the voltage measurement, it would appear that instrumental error in measurement of voltage and power should not exceed 0.6 per cent when a good voltmeter is used or about 0.2 per cent when a potentiometer is used.

The dynamometer, which is used as a null instrument, must have a sensitivity high enough so that Eq. 3 can be satisfied to the desired accuracy by adjusting the auxiliary yoke magnetization. This sensitivity has been increased as much as seems feasible, being in fact now as great as that of the better d-c. galvanometers. If the error due to adjustment of the yoke magnetization is to be kept under ± 1 per cent, the deflection of this instrument must not exceed ± 1 mm. Since the deflection can be kept under 0.4 mm. the total instrumental error can be kept to 1 per cent when a voltmeter is used and can be much less than this when a potentiometer is used.

The only remaining factors influencing the inherent accuracy are the coil constants and the cross-section area of the specimen. These coil constants are known with a very high degree of accuracy by *absolute* measurements. The coil constants involved can best be appreciated from an inspection of Eqs. 7a and 9 upon which the calculation of the core loss from the instrument readings is based.

The number of turns per centimeter in the magnetizing winding, n, is known to about ± 0.05 per cent. The total number of turns in the B secondary, N, is known exactly by the use of a mechanical counter which reads to $\frac{1}{10}$ of a revolution and since all turns must be completed there can be no error in the turn count. The cross-section, a, may be calculated from the observed weight and length of the sample by means of the formula

$$a = \frac{w}{l\rho}$$

where w = weight in grams,
l = length in centimeters, and
ρ = density of the sample.

The cross-section, a, can be determined to 0.1 per cent by this method if the quantities involved are measured carefully. If this is not done an appreciable error can arise from this cause. If the A.S.TM. Standard Methods of Test for Magnetic Properties of Iron and Steel (A 34 - 41)[16] is adhered to in regard to the value of ρ, which is taken as 7.5 for silicon steels containing more than 2 per cent silicon and 7.7 for lower silicon content, an appreciable absolute error may result. (The cross-section area can also be determined from width and thickness measurements and this method is usually employed where long strips of steel are to be tested.) $B_{\max.}$ is determined by E_{avg}. which has already been considered as an instrumental error.

The constants of the coils which are used in connection with the null dynamometer in determining when the condition of Eq. 3 is satisfied, are calibrated by an absolute method against a standard absolute field. The calibration can be made to ± 0.02 per cent and the standard field is known by calculation from the very accurately fixed dimensions, as well as by actual measurements to better than ± 0.01 per cent.

All of the factors involved show that the inherent absolute accuracy of core loss measurements made by the single strip method is within ± 1 per cent even in the case of the mill testers with their relatively less accurate voltmeter.

In order to attain this accuracy all instrument circuit resistance must be kept within the required limits, the voltmeter calibration must be checked periodically, the wattmeter must be calibrated for full deflection daily, the precision commutator used with the voltmeter must be kept clean in order to maintain the voltmeter circuit resistance (an oscillograph is connected to this commutator at all times and the slightest faulty contact will show up at once), and the weight, and length, and density of the sample must be carefully determined.

PRECISION OF MEASUREMENTS

The precision or reproducibility of the method is influenced by two factors; the skill of the observer and the presence of electrical, mechanical, or magnetic disturbances. The majority of personal errors arise from poor lighting, some distracting influence, or poor judgment, but these may be kept to a minimum after some experience. Even an experienced observer, however, occasionally makes an error in reading an instrument.

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[16] 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 121.

The electrical disturbance most likely to be troublesome is a fluctuation in the voltage or frequency of the power supply. Some difficulty has also arisen from the leakage of current into the null dynamometer circuit.

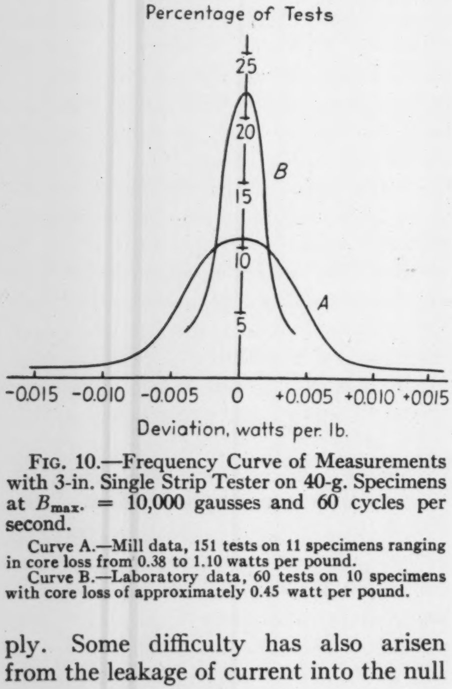

FIG. 10.—Frequency Curve of Measurements with 3-in. Single Strip Tester on 40-g. Specimens at $B_{max.}$ = 10,000 gausses and 60 cycles per second.

Curve A.—Mill data, 151 tests on 11 specimens ranging in core loss from 0.38 to 1.10 watts per pound.
Curve B.—Laboratory data, 60 tests on 10 specimens with core loss of approximately 0.45 watt per pound.

Mechanical disturbance which results chiefly from heavy vibration of the building may influence both the suspension type wattmeter and the null dynamometer. Except for an occasional very severe shock which shifts the zero of the instruments appreciably, vibration has been virtually eliminated by the use of a suitable mounting. The difficulty may also be overcome by increasing the ratio of deflection to disturbance by about fifty times by inserting a suitable amplifier in the null dynamometer circuit.

Strong alternating magnetic fields of 60-cycle frequency must be avoided in close proximity to the sensitive measuring instruments or the tester coils since the total power being measured is often only 0.01 watt so that if an accuracy of 1 per cent is to be obtained, the power must be measured to closer than 0.0001 watt, that is, to one tenth of a milliwatt.

Constant magnetic fields or fields varying at any rate other than 60 cycles per second, cannot exert any influence on the measurements unless, of course, they act upon the specimen directly to produce an additional magnetization superimposed on the sixty cycle magnetization. This condition cannot arise, however, if the source of all magnetic fields is kept at least 10 ft. away from the tester coils. So far as the instruments are concerned they are unaffected by such fields unless they become badly contaminated with magnetic material. All necessary precautions were taken to prevent this during the building of the instruments and with reasonable care in use they should remain free of magnetic contamination.

The precision of this method is illustrated by the frequency curves of Fig. 10 which show that the precision obtained in the laboratory is somewhat better than that in the mill, a result to be expected in view of the use of a more precise instrument in the laboratory and of the fact that the mechanical and electrical disturbances were smaller in the laboratory tester. Even so, of the 151 tests made at the mill, which include specimens ranging in core loss from 0.38 to 1.10 watt per pound, 76 per cent lie within ± 1 per cent of the core loss value for all specimens, except for two samples which were less than 0.400 watt per pound, in which case the deviation is approximately 1.25 per cent. In the 60 tests made in the laboratory, the core loss for all samples was approximately 0.450 watt per pound and 98.3 per cent of the observations lie within ± 1 per cent, whereas all lie within 1.1 per cent.

These data indicate that even under relatively unfavorable conditions in a mill, a high degree of precision is obtained, which is especially notable because the portion of the sample in which the core loss was measured weighed only about 40 g. and the total power measured was about 0.040 watts in the most unfavorable case, hence the error of measurement is less than 0.0004 watts. Furthermore, a specimen one third as large can be measured in the same tester with nearly the same precision as that shown in the frequency curves.

Conditions are quite different in the standard Epstein test using 10 kg. of iron with a total core loss of 10 watts to be measured. In spite of this favorable condition brought about by the large mass of the sample, the precision of the Epstein readings is only moderate.

CORRELATION OF SINGLE STRIP AND EPSTEIN CORE LOSS DATA

In making any comparison between data obtained by two different test methods it is necessary to compare results obtained on exactly the same material by the two methods. This may seem to be an obvious statement, but nevertheless it is a matter often overlooked, either completely or partially, with the result that very misleading conclusions may be reached. Early attempts to correlate core loss data obtained on the whole sheet tester with those obtained by the Epstein method failed for this reason. It must be remembered that the whole sheet tester and the various single strip testers were designed to measure the magnetic properties of the middle 40 per cent of that portion of the specimen bridging the ends of the yoke. The Epstein sample must be cut from this portion of the specimen and, if a representative comparison is to be made, must include all of this material. If, as in the case of the first attempts at correlation, the entire sheet is cut up for the Epstein test, a true comparison is not obtained unless the sheet is uniform in magnetic properties from end to end. This is not always the case, but from a number of exploratory tests it appeared that the core loss of the middle portion of the sheet was the average of that for the entire sheet. This was believed to be always the case and therefore correlation of Epstein data on the entire sheet with whole sheet tester results on 28 in. of the length of the sheet was attempted. This failed utterly and it was found that firstly, the center section of the sheet was not always representative of the entire sheet, and secondly, that the effect of cutting the sheet into strips for the Epstein test was much larger and much more variable than had heretofore been realized. Later correlation tests were planned so that these variables would not enter and vitiate the results.

There are two ways that these variables may be eliminated: the sheet may be cut into strips $1\frac{3}{16}$ in. wide for the Epstein test before the box anneal and the tests will then be performed on strain-free material; or it may be cut into strips after the box anneal but before making the test in the whole sheet tester, the measurements in the two tests being then made on material in a comparable state of strain, since the additional strain introduced by cutting the strips into proper lengths for the

Epstein test is negligible. When tests by each method are made in this manner on the same material, the true agreement is shown. Since the core loss as measured by the Epstein test is influenced to some extent by cold working during cutting, which effect is absent in the whole sheet tester, as used in production, these tests do not provide a strict correlation between the two methods. If the results obtained with the whole sheet tester are to be interpreted in terms of Epstein values (a procedure that is desirable at present because experience or design of electrical equipment is based largely, though somewhat empirically, upon the data obtained by the Epstein test) it is necessary to add a representative average effect of cutting to the whole sheet test result. Some data have been obtained with the aid of one of the 3-in. single strip tester (for which we are indebted to G. Weber Smith, Magnetic Testing Engineer) which show that the effect of cutting varies with the length of time the shear blades have been used. For this purpose strips of silicon steel sheet were cut to a width of $2\frac{1}{2}$ in., annealed to relieve them of strain and tested for core loss at $B_{\text{max.}} = 10{,}000$ gausses and at 60 cycles per second. These strips were then trimmed at each edge and sheared into Epstein size $1\frac{3}{16}$-in. strips and retested. Data were obtained on a number of samples in this manner on one grade of material at various times after the shear blades had been installed. (The shears are in practically continuous use for shearing all grades of electrical sheet for Epstein tests and the period covered by the tests for effect of shearing on core loss is less than the usual replacement period for the shear knives.) These experiments should of course be repeated for all the usual grades and gages of material to be tested in whole sheet form in order to arrive at the best average factor to be applied in each case. The results of the first tests on the effect of shearing on core loss are summarized in Table I and show that even with reasonably good knives shearing may increase the core loss by as much as 15 per cent.

TABLE I.—OBSERVED INCREASE IN CORE LOSS DUE TO SHEARING IN PREPARATION OF SPECIMENS FOR EPSTEIN TEST.

Length of Time Shear Knives Had Been in Use, hr.	Increase in Core Loss, per cent
11	6·3
27	8·1
37	10·6
53	15·1

TABLE II.—COMPARISON OF MEASUREMENTS MADE SIMULTANEOUSLY ON ALL THE STRIPS.

Each 108 in. long by $1\frac{3}{16}$ in. wide, cut from a single sheet with these strips (*a*) alongside each other in the 36 in. wide tester (*b*) later cut to standard length and grouped in the Epstein test.

Material		Number of Sheets Tested	Percentage by Which Epstein Test Is Higher	
			Minimum	Maximum
Strips sheared, then annealed at 1950 F.		2	4·0	6·0
Strips sheared, from fully annealed sheet; shearing strains not removed:	lot 1	2	1·8	2·1
	lot 2	4	−0·1	+2·3
	lot 3	6	1·2	3·8
	lot 4	10	1·7	7·6

Subject to these comments on the effect of shearing, the correlation data are presented in Table II. These data show that nearly all the data fall in the range −0.1 to 7.6, a spread of 7.7 per cent which is quite within reason for the correlation between an absolute test method and a nonabsolute one, especially in consideration that the Epstein method has well-known errors which vary with the magnetic properties of the sample to this extent. Of course on the basis of these few data it cannot be definitely stated that the test results will always fall within this range of values, but the difference between the Epstein results and the whole sheet results is so small that it is probable that this will be the case at least for magnetic material that is not materially better than that on which the data were obtained.

SUMMARY

Electrical sheet is rated chiefly by the energy loss under cyclic magnetization. A method is described which evaluates accurately this important characteristic in a simple manner in terms of absolute electrical measurements; it lends itself to power loss measurement in whole sheets as well as in small specimens comparable in size with a single strip of an Epstein core. In spite of the necessarily small magnitude of power loss in such a small specimen the error, which amounts to less than 0.0005 watt, may be consistently limited to about 1 per cent in routine testing. The precision or relative accuracy is likewise high as is shown by frequency curves of a number of repeated measurements on the smallest of the four single-strip testers which have been in use for some time.

In this method the ideal conditions of measurement can be approximated as closely as is desired. These conditions are a homogeneous magnetizing field, H, and a pure harmonic time-variation of the magnetic flux throughout the specimen. The homogeneous field of an infinite solenoid can be realized in a relatively short solenoid by completing the magnetic circuit of this coil and its core through a laminated iron yoke which is magnetized by separate windings not connected in the power measuring circuit. The harmonic time-variation of the flux can be achieved by superimposing and compensating magnetizations of higher order odd harmonics by means of these separate yoke windings. The adjustment in amplitude and phase of these two auxiliary magnetizations is effected through a null method, hence is independent of the calibration of the indicating instrument and is capable of great sensitivity. In the case of the first of these compensations, the null method consists of balancing an electromotive force inductively derived from the magnetizing current against the emf. also inductively derived from the field of the short solenoid. The second compensation to adjust the time variation of the flux is effected by a null method based on measuring the higher odd harmonics of the emf. induced in the secondary wound about the specimen; this measurement, which is made with the same dynamometer used in the field homogeneity adjustment, is simplified by the well-known property of a dynamometer of responding only to that frequency used in energizing its field, thus making possible the adjustment of each harmonic separately. When these field conditions for the specimen have thus been established, the maximum flux density in the specimen is measured by means of a d-c. voltmeter and a precision mechanical rectifier, and the corresponding core loss is measured by means of a special high-sensitivity wattmeter.

Since the Epstein method is the conventional test for core loss, some comparative data are reported to illustrate the degree of agreement of this empirical method with the absolute method described in this paper. These measurements were made on the identical samples of sheet silicon steel with special precautions to insure that the material was in exactly the same physical condition when tested by each method. The Epstein results deviate by from -0.1 to $+7.6$ per cent from the corresponding values; this is not a large range consid-

ering that in the Epstein method there are well-known errors of this magnitude which vary with the magnetic properties of the material and the make-up of the core as tested. The difference between the Epstein test, as usually carried out, and this absolute method, as applied to a whole sheet, is however considerably greater than the frequency figures indicate, because the mere shearing of the material into Epstein strips may, and frequently does, increase the core loss by as much as 15 per cent.

Acknowledgment:

The development has been aided by the cooperation and advice of a number of persons. I am especially indebted to my colleagues M. E. Berley and D. S. Miller, to the Chairman and members of the Electrical Sheet and Strip Development Committee of the Carnegie-Illinois Steel Corp.; and to the personnel of the magnetic testing laboratories at the Vandergrift plant, Carnegie-Illinois Steel Corp., and at the Cuyahoga Works, American Steel and Wire Co. It is also a pleasure to acknowledge the invaluable aid of W. N. Goodwin, Jr., and A. H. Wolferz of the Weston Electrical Instrument Co. in developing the dynamometer to its present state of perfection.

DISCUSSION

MR. THOMAS SPOONER.[1]—Several months ago a number of the members of Committee A-6 on Magnetic Properties were invited to visit the Vandergrift Plant of the Carnegie-Illinois Steel Co., and to see two of Mr. Shenk's devices in actual operation. We were very much impressed, and I took it upon myself not long ago to urge Mr. Shenk to prepare a paper, for presentation before the Society, describing his new developments, and this paper which you have just heard is, perhaps, partly the result of my suggestion. For many years I have felt that such a device would be very useful, but it has been a very difficult problem, which probably accounts for the fact that such a tester has not been developed long ago. The advantages are obvious—a saving in test material, probably a saving in testing time and, of course, the elimination of the effect of strains, as Mr. Shenk has brought out. Many years ago we came to the conclusion that increases in core loss for a 1 in. wide strip, due to punching, would be of the order—on the average—of 10 per cent which checks pretty closely with Mr. Shenk's figures. Also there are corresponding decreases in permeability for certain ranges of induction.

We can be very grateful to Mr. Shenk for this development. Some day Committee A-6 may find it advisable to adopt a method—either this method or a similar one—as a standard method for testing the magnetic properties of sheet material. It may be some time before the accuracy and suitability of the method is sufficiently proved so that we would care to take that step, but I think it is a probability.

I am wondering whether Mr. Shenk has considered modifying his tester so that it can be used to determine the magnetic properties of strip material passed continuously through the coils. In that way one could follow variations and would have a very good tool which could be used successfully, I believe, by the mills in studying inhomogeneities and in effecting greater uniformity of material.

MR. J. P. BARTON.[2]—This paper represents an advanced link in the chain leading towards improved methods of measurement of the magnetic characteristics of flat rolled steels, primarily silicon steel. It certainly represents an advancement in the art of measuring core loss and a-c. permeability, probably to a greater degree of precision than heretofore. Of particular importance is the fact that this is an absolute method, whereas other methods employed to date, with the exception of Burrough's permeameter, are not of the absolute type. By this new method it is possible to measure strips ranging from approximately 1 in. wide up to 36 in. or wider, and it offers the distinct advantages of being able to measure the properties of sheets and material in coils without destroying that material. With the existing methods of test it is necessary to cut strips, which represents an economic loss of that material.

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[1] Chairman, A.S.T.M. Committee A-6 on Magnetic Properties; Manager, Engineering Laboratories and Standards Dept., Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[2] Manager, Bureau of Electrical Sheet Sales, Carnegie-Illinois Steel Corp., Pittsburgh, Pa.

Another advantage of Mr. Shenk's method is that it is a powerful tool in the development laboratory, as it is possible to measure accurately single strips before and after, for instance, a heat treatment, thus showing the effect of that treatment on one specimen strip. Heretofore, equipment for measuring core loss and permeability required a number of strips, on which it was necessary to assume that all of the strips had uniform characteristics. In some cases this assumption led to marked errors, as all the strips would not respond in the same manner to whatever treatment was being applied. With Mr. Shenk's method it is now possible to measure an entire sheet of silicon steel by shearing the sheet into strips of convenient width and length, thus showing the variation in magnetic properties from area to area in the sheet.

As Mr. Spooner has just mentioned, it would seem that this equipment has possibilities towards measuring, at least semicontinuously, the magnetic properties of electrical strip in coils. We can look forward to this possibility as a means of quality control on a simple basis, similar, for instance, to apparatus now used in indicating the thickness of cold reduced strip as it comes off the cold reduction mills.

Proposed tentative standard methods have been under consideration which make it possible to improve the degree of accuracy in measuring core loss and a-c. permeability simultaneously. These methods have been under development in Committee A-6 and should help towards narrowing the gap between Mr. Shenk's apparatus and the tentative and standard methods sponsored by Committee A-6.

Mr. Shenk is to be congratulated on the development of this absolute method of test. It has taken a number of years for this very complete and thorough investigation to bring this apparatus to the precision that he has obtained.

MR. J. Q. ADAMS.[3]—I am particularly interested in knowing how narrow a strip can be readily used in this type of apparatus, and whether Mr. Shenk has worked with the narrow strip steels. I note that his paper deals primarily with the wider sheets.

MR. W. E. SHENK[4] (*author's closure*).—I want to thank Mr. Spooner for the great encouragement he has given me in preparing the paper. His enthusiasm as to the possibilities of the new test method when he and some of his associates on Committee A-6 visited the Vandergrift Plant of the Carnegie-Illinois Steel Corp. was very largely responsible for the presentation of a description of the method at this time.

The advantages pointed out by Mr. Spooner, namely, saving in test material, probable saving in testing time, and elimination of the effect of strains, were, together with the possibility of testing the identical material to be used in the magnetic core of an electrical machine the chief objectives sought when the development of a new test method for core loss and a-c. permeability was undertaken several years ago. It is indeed gratifying to have Mr. Spooner, Chairman of Committee A-6 on Magnetic Properties, point out that the goal has been achieved.

Both Mr. Spooner and Mr. Barton have raised the question of measuring the magnetic characteristics of electrical strip continuously. The accurate measurement of core loss with the strip specimen in motion was not considered because to do so would have unduly complicated the apparatus and would have multiplied many times the difficulty of obtaining suitable instruments. A relative and at least qualitative continuous measurement can be obtained however, by adjusting the yoke magnetization and flux density in the specimen at the start of a coil of electrical strip and then as the strip advances continuously, observing the changes in the wattmeter reading which will indicate a variation between high and low loss in the strip. A reference upon which to base these relative variations can be established by either cutting off the end of the strip for an accurate core loss measurement in the strip tester or by stopping the strip for a brief interval and measuring its core loss accurately. This method has been tried only experimentally so far, but it shows promise.

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[3] Metallurgist, General Electric Co., Schenectady, N. Y.

[4] Research Engineer, Research Laboratory, United States Steel Corp., Kearny, N. J.

Mr. Barton has brought out an additional advantage of the new test method when used for a narrow strip, that is, the possibility of measuring the magnetic properties of a single strip before and after any desired treatment. In this way it is possible to ascertain the optimum treatment for a given magnetic material or to determine the properties of an experimental material available only in very small quantity. Thus a great economy in development costs can be effected.

Mr. Barton has emphasized the fact that the new test method is absolute and had pointed out that, with the exception of the Burrough's permeameter, it is unique in this respect. Since however, the Burrough's permeameter is a d-c. test, it is hardly comparable with the present method which is therefore the only absolute a-c. test.

Mr. Adams asks how narrow a strip can be measured. The testers are designed in several sizes to cover a range of from 1 in. wide to 36 in. wide strip. The method was worked out first on narrow specimens and a tester for pieces of strip 1 to 3 in. in width and at least 10 in. long was built. Duplicates of this tester were built for the Vandergrift Plant of the Carnegie-Illinois Steel Corp. and the Cuyahoga Works of the American Steel and Wire Co. where they are regularly used in production testing as well as in development work. The larger testers are primarily designed for production testing, although they too can be used to some extent in development.

Another possible field of usefulness of the tester is the measurement of core loss and permeability under superimposed d-c. and a-c. magnetization. This application, which is important in the communications field, will be discussed in a future paper.

A STUDY OF THE CHEMICAL, PHYSICAL AND MECHANICAL PROPERTIES OF PERMANENT MOLD GRAY IRON

By Richard Schneidewind[1] and Edward C. Hoenicke[2]

Synopsis

Determinations have been made of the mechanical properties of permanent mold irons which are irons of gray iron composition cast in water-cooled metal molds and fully annealed to give a structure of alpha ferrite and graphite. The results show properties considerably higher than would be expected from material of such microstructure.

It is significant that although fully annealed, the properties of permanent mold irons vary inversely as the section size of the casting; that is, irons of small section size are stronger than those of heavy section size.

Permanent mold iron castings are of a modified gray iron composition cast into permanent air-cooled or water-cooled metal molds. The metal molds are given coatings which prevent the castings from adhering to the mold and reduce the thermal shock when the molten metal strikes the mold. If applied in two layers, the original or base mold coating is a refractory clay on which is superimposed the second coating, which is graphite in the form of carbon smoke; some plants employ a single coating.

Some castings are produced in stationary molds, while water pipe made by the De Lavaud process is cast centrifugally in rotating molds. All permanent mold gray iron castings are given a complete anneal. One such annealing treatment consists of heating to 1575 F. in $1\frac{1}{2}$ hr., holding at that temperature for $\frac{1}{2}$ hr., and cooling slowly through the critical range in $1\frac{1}{2}$ hr. The microstructure after casting consists of lacy eutectiform graphite, ferrite, and a small amount of pearlite. No massive carbides are present. After the annealing treatment, the matrix is completely ferritic.

Most metallurgists would assume from this microstructure that the mechanical properties are low, in the order of magnitude of 18,000 psi. in tensile strength. This misconception arises from the fact that when ordinary gray iron, sand cast, is given a full annealing treatment to a ferritic matrix, the tensile properties are in that range. Many tests made on permanent mold irons and on permanent mold castings have indicated much higher mechanical properties. This investigation was started in order to determine more of the properties of permanent mold irons which might be of assistance to designing engineers.

Summary of Conclusions

The results of the present investigation have led to the following conclusions:

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[1] Associate Professor of Metallurgical Engineering, Department of Chemical and Metallurgical Engineering, University of Michigan, Ann Arbor, Mich.

[2] Eaton Erb Foundry Division, Eaton Manufacturing Co., Detroit, Mich.

1. Permanent mold irons have a microstructure consisting of ferrite and graphite. Faint traces of eutectoid carbides are sometimes present.

2. These irons, although possessing the structure noted above, are sensitive to section size, that is, the mechanical properties vary with section size.

3. The smaller the section size, the finer is the pattern of eutectiform graphite. In large sections, the center of the casting may contain some fine flake graphite.

4. The Brinell hardness is about the same for all section sizes investigated, namely, 153 to 168. Permanent mold castings can be produced, with modifications in procedure, to a Brinell hardness of 190 or higher to 207 and yet have a microstructure of ferrite and graphite. For most applications, however, the softer and more machinable material is demanded and for this reason this investigation was conducted on castings representing a greater proportion of consumer tonnage.

5. Table I summarizes the properties obtained for castings varying in diameter from 0.5 in. to 2.0 in.

Procedure and Results

The test bars were cast to such a size that machining off the surface skin left them with 2, 1.2, 0.875, and 0.5-in. diameters and in 18 to 24-in. lengths. The bars were not taken from any one specific heat but were prepared from production heats over a period of several months.

TABLE I.—PROPERTIES FOR PERMANENT MOLD CASTINGS 0.5 TO 2.0 IN. IN DIAMETER.

Section diameter, in.	0.5	0.875	1.2	2.0
Tensile strength, psi	52 267	34 142	32 050	22 533
Tensile Modulus of Elasticity, psi	19 530 000	17 570 000	13 600 000	11 980 000
Brinell hardness	153	163	168	164
Compressive strength, psi	124 342	112 017	119 583	99 783
Torsion, yield shear, psi	......	23 160	19 208	20 208
Torsion, rupture shear, psi	......	43 750	42 186	36 483
Torsion modulus of elasticity, psi	......	7 435 000	6 747 000	6 310 000
Transverse breaking load, lb.	606	1 665	2 670	8 200
Transverse deflection, in.	0.178	0.166	0.283	0.240
Modulus of rupture, psi.	98 950	75 933	70 783	62 700
Bending modulus of elasticity, psi.	19 600 000	16 940 000	14 380 000	14 870 000
Plastic work, per cent	57.1	40.1	37.7	30.5
Endurance limit, psi.	32 000	20 000	19 500	13 500
Endurance limit, notched bars, psi.	<15 000	15 000	......	13 000
Heat-treated, tensile strength, psi.	82 433	50 250	38 783	32 242
Heat-treated, tensile modulus of elasticity, psi	19 200 000	17 400 000	13 360 000	10 950 000
Growth, increase in volume, per cent per cycle	0.133	0.170	0.180	0.330

Chemical Analysis:

It was entirely possible that no two bars were of the same heat but, inasmuch as the foundry practices were fairly uniform, one bar was taken at random for chemical analysis which proved to be as follows:

Total carbon, per cent	3.52
Graphitic carbon, per cent	3.41
Combined carbon, per cent	0.11
Silicon, per cent	2.55
Manganese, per cent	1.01
Sulfur, per cent	0.086
Phosphorus, per cent	0.215

This value was consistent with the plant analyses made during this period. If the value, carbon plus one-third silicon is computed, the result is 4.37 per cent. This iron is, therefore, of eutectic composition. Compared to normal gray irons, it is very high in silicon and would be suitable only for very small section sizes when cast in sand.

Tensile Properties:

Specimens were machined from each size bar for tension tests. The diameter of the reduced section was 0.75 in. for the 2.0-in. and 1.2-in. bars; 0.5 in. for the 0.875-in. bars, and 0.375 in. for the 0.5-in. bars. It has been found in tests at the University of Michigan over a period of years that the standard 0.79-in. diameter reduced section results in a large percentage of breakage in the threaded portion. The other dimensions of the tension specimens were as specified in A.S.T.M. Tentative Methods of Tension Testing of Metallic Materials (E 8 - 40 T).[3] Four bars of each category were broken and the results averaged. Modulus of elasticity was computed from stress-strain diagrams. The results are given in Table II.

TABLE II.—TENSILE PROPERTIES OF PERMANENT MOLD IRONS.

Bar Size, in.	Tensile Strength, psi.	Modulus of Elasticity, E, psi.
2.0	22 533	11 980 000
1.2	32 050	13 600 000
0.875	34 142	17 570 000
0.5	52 267	19 530 000

TABLE III.—TENSILE PROPERTIES OF 2.0-IN. DIAMETER BARS.

Each value is the average of three consistent figures.

Reduced Section Diameter, in.	Tensile Strength, psi.
1.25	22 180
0.75	22 533
0.5	22 892
0.375	22 700

In order to avoid criticism of the use of a reduced section of 0.75 in. for the 2-in. diameter bars, three additional sets were machined from the 2-in. bars having reduced sections of 1.25 in., 0.5 in., and 0.375 in., respectively. The tensile values obtained on these specimens are given in Table III; each value is the average of three consistent figures. It may be seen that the diameter of the test specimen does not appreciably influence the tension test values obtained.

TABLE IV.—BRINELL HARDNESS OF PERMANENT MOLD IRONS.

Averaged values.

Bar Size, in.	Brinell Hardness
2.0	164
1.2	168
0.875	163
0.5	153

TABLE V.—COMPRESSIVE STRENGTH OF PERMANENT MOLD IRONS.

Average of at least four individual determinations.

Bar Size, in.	Compressive Strength, psi.
2	99 783
1.2	119 583
0.875	112 017
0.5	124 342

Brinell Hardness:

Brinell hardness measurements were made on many samples of each size bar. The results were consistent and the averaged values are given in Table IV.

It may be seen that there is little appreciable difference in Brinell hardness between bars of different diameters.

Compressive Strength:

Cylinders were prepared for compressive strength tests. These cylinders were the full diameter of the bars and the height was twice the diameter. The results are given in Table V.

All of these values are the average of at least four individual determinations.

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[3] 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 453.

Torsion Properties:

The torsion tests were conducted in an Olsen torsion testing machine. The specimens were tested with full diameter and length. The gage length used for computation was 8.25 in. and the stress was applied in inch-pounds. The 0.5-in. diameter bars could not be tested because they did not fit the machine. The results of the tests are given in Table VI.

TABLE VI.—TORSIONAL PROPERTIES OF PERMANENT MOLD IRONS.
Averaged values.

Diameter of Bar, in.	Shear Stress at Yield Point, psi.	Shear Stress at Rupture, psi.	Torsional Modulus of Elasticity, E_s, psi.
2.......	20 208	36 483	6 310 000
1.2.....	19 000	42 186	6 747 145
0.875...	23 160	43 750	7 435 000

These values are shown graphically in Fig. 1, where the diameter of the bar is plotted against the torsional properties.

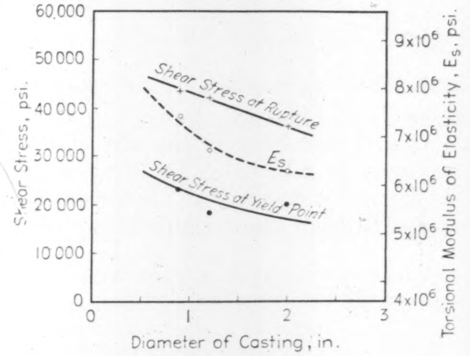

FIG. 1.—Relationship Between Torsion Properties and Diameter of Section.

For purposes of comparison a few values of torsional modulus of elasticity of other materials are given here:

Steel, psi.	11 500 000
Gray cast iron, psi.	6 000 000
Malleable cast iron, psi.	7 500 000 to 9 500 000
Aluminum, psi.	3 600 000
Brass, psi.	5 400 000
Bronze, psi.	6 000 000

Bending or Transverse Properties:

The physical properties in bending were determined in an Amsler hydraulic testing machine. The 0.5-in. bars were broken on 8-in. supports, the 0.875-in. bars on 12-in. supports, the 1.2-in. bars on 18-in. supports, and the 2.0-in. bars on 24-in. supports. The load was applied midway between the supports and the deflection was measured directly below the point of loading by means of a lever and an Ames dial. Deflection was measured to 0.0001 in. At least four bars of each size were broken and stress-strain diagrams were drawn. The moduli were computed where the load equalled 50 per cent or less of the breaking load. The averaged values are shown in Table VII. The breaking energy in inch-pounds was computed for each bar from the stress-strain diagrams by obtaining the area under the stress-strain curves. The averaged values are also given in Table VII.

TABLE VII.—TRANSVERSE PROPERTIES OF PERMANENT MOLD IRONS.
Averaged values

Diameter of Bar, in.	Breaking Load, lb.	Deflection, in.	Modulus of Rupture, psi.	E, Bending Modulus, psi.	Breaking Energy, in-lb.	Plastic Work, per cent
2.......	8200	0.240	62 700	14 872 000	1177	30.5
1.2.....	2670	0.283	70 783	14 378 300	482	37.7
0.875...	1665	0.166	75 933	16 940 000	176	40.1
0.5.....	606	0.178	98 950	19 600 000	72.3	57.1

When a bar of cast iron is broken, a certain amount of the energy released is recoverable. The remainder is plastic. From the modulus of elasticity and the breaking load the amount of elastic work may be computed by obtaining the area of a right-angle triangle whose height is the breaking load and whose hypotenuse is an extension of Young's modulus line. If this is subtracted from the total breaking energy, the amount of plastic work is obtained which represents the permanent set. The amount of plastic work may be expressed as a percentage of the total work energy required to break the bar and is a measure of the toughness of the iron. These values are given in the last column of Table VII. They show that the smaller the diameter the greater the toughness or plasticity of the iron.

In reviewing the values in Table VII, it is obvious that breaking load deflection and breaking energy cannot be compared from bar to bar to give any reasonable comparison of a specific property of the iron.

This is true because the size of the specimen is different. The transverse or flexural strengths can, however, be compared from the modulus of rupture.

TABLE VIII.—FATIGUE TESTS ON PERMANENT MOLD IRONS.

NOTE.—Italicized values mean the specimen broke.
Va`ues not italicized indicate the specimen did not break.

0.500 in. Diameter, Tensile Strength = 52,267 psi.		0.875 in. Diameter, Tensile Strength = 34,142 psi.		1.200 in. Diameter, Tensile Strength = 32,517 psi.		2.00 in. Diameter, Tensile Strength = 22,533 psi.	
Stress, psi.	Revolutions to Failure	Stress, psi.	Revolutions to Failure	Stress, psi,	Revolutions to Failure	Stress, psi.	Revolutions to Failure
PLAIN SPECIMENS							
28 300	53 977 000	9 440	3 055 000	19 100	39 147 000	12 850	41 474 000
29 600	17 845 000	13 200	17 798 000	20 650	*3 935 000*	13 970	*6 720 000*
31 200	23 206 000	17 080	13 001 000	22 150	*436 000*	14 800	*212 000*
31 850	55 978 000	18 960	28 825 000	23 0 0	*400 000*	15 180	*13 000*
32 600	*4 000*	19 700	23 093 000	26 900	*364 000*	......	....
33 360	*2 000*	20 220	*5 739 000*	......	....	......	....
......	....	23 800	*4 030 000*	......	....	......	....
......	....	23 850	*4 847 000*	......	....	......	....
NOTCHED SPECIMENS							
15 120	*4 850 000*	13 490	29 847 000	13 250	*6 906 000*	9 820	20 980 000
19 680	*559 000*	14 980	13 533 000	13 890	81 926 000	11 730	20 448 000
20 850	*154 000*	15 300	*4 233 000*	14 760	*677 000*	13 620	*755 000*
22 700	*768 000*	15 970	*1 078 000*	15 400	107 186 000	14 010	*922 000*
......	....	16 900	*4 046 000*	17 100	*430 000*	15 130	*35 000*
......	....	21 200	*39 000*	......	....	......	....
......	....	24 400	*10 000*	......	....	......	....

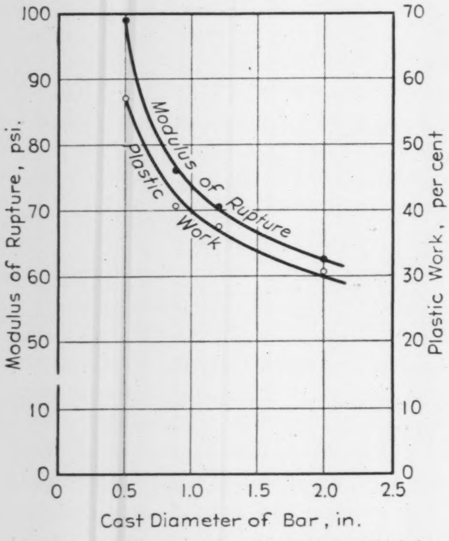

FIG. 2.—Influence of Section Size on Modulus of Rupture and on Plastic Work of Permanent Mold Irons in Transverse Bending.

TABLE IX.—ENDURANCE PROPERTIES OF PERMANENT MOLD IRONS.

Diameter, in.	Endurance Limit, psi.	Tensile Strength, psi.	Ratio of Endurance Limit to Tensile Strength, per cent
STANDARD BARS			
0.5.......	32 000	52 267	61.3
0.875.....	30 000	34 142	59.6
1.2.......	19 500	32 517	60.0
2.0.......	13 500	22 533	60.0
NOTCHED BARS			
0.5.......	<15 000	52 267	....
0.875.....	15 000	34 142	44.0
1.2.......	?	32 517	....
2.0.......	13 000	22 533	57.5

It can be seen that the modulus of rupture increases about 50 per cent as the diameter of the bar is reduced from 2.0 in. to 0.5 in. Likewise, the bending modulus increases about 50 per cent as the diameter of the bar is reduced from 2 in. to 0.5 in. The plasticity of the iron has been discussed before.

These results are presented graphically in Fig. 2, where the diameter of the bar is plotted against modulus of rupture and the percentage of plastic work.

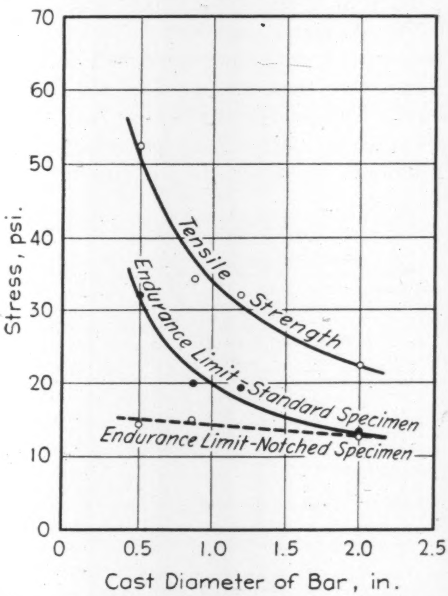

FIG. 3.—Influence of Section Size and Notch on Endurance Limit of Permanent Mold Irons—R. R. Moore Machine.

Fatigue Properties:

Standard fatigue specimens were prepared from each diameter bar and were tested on the R. R. Moore type of rotating-beam machine.

In addition, another series of specimens from each diameter bar was prepared, which contained a V-notch in the center of the specimen. The results of the individual tests are given in Table VIII and endurance limit is presented in Table IX.

The results would indicate that endurance limit decreases with increased size of bar. In Table IX the tensile strength values have also been presented along with the ratio of endurance limit to tensile strength. In the case of the standard fatigue bars, the results indicate a somewhat high ratio of endurance limit to tensile strength normal for cast irons. This ratio may vary from 43 to 58 per cent.[4] In the case of these permanent mold irons, the endurance limit proved in every case to be very close to 60 per cent of the tensile strength. No explanation can be given at this time for this abnormally high ratio.

TABLE X.—COEFFICIENT OF EXPANSION OF PERMANENT MOLD IRONS.

Temperature Range, deg. Fahr.	Average Coefficient of Expansion, in. per inch per deg. Fahr. $\times 10^6$				Instantaneous Coefficient of Expansion for S.A.E. 1015 Steel, in. per inch per deg. Fahr. $\times 10^6$
	0.5-in. Diameter	0.875-in. Diameter	1.2-in. Diameter	2.0-in. Diameter	
70 to 200	6.65	6.65	7.22	6.45	....
70 to 400	6.74	6.74	7.11	6.66	....
70 to 600	6.90	6.90	7.19	6.86	....
70 to 800	7.11	7.05	7.82	7.08	7.83
70 to 1000	7.31	7.16	7.45	7.29	8.02
70 to 1200	7.58	7.27	7.71	7.51	8.36
70 to 1300	7.69	7.36	7.86	7.62	....

It would appear that the notched-bar fatigue properties are practically constant at between 13,000 and 15,000 psi., regardless of the original cast diameter of the bar. Difficulty was experienced in obtaining consistent results on the notched bars.

The endurance limit on standard bars or notched bars and the tensile strength are shown graphically in Fig. 3, where these properties are plotted against the diameter of the bar as originally cast. The test specimens were of standard dimensions.

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[4] "Alloy Cast Irons," p. 86, Am. Foundrymen's Assn. (1939).

Coefficient of Expansion:

Specimens were machined 4 in. long and $\frac{3}{8}$ in. in diameter. They were placed in a dilatometer built in accordance with the A.S.T.M. specifications. The results of the test are given in Table X. These values are the average values between room temperature and the temperature indicated. The instantaneous coefficient of expansion of S.A.E. 1015 steel is included in this table for purposes of comparison.

Impact Tests:

In view of the fact that there is no generally acceptable standard for impact bars in the cast iron field, test specimens were made up with the following dimensions: The 2-in. and 1.2-in. bars were machined to a diameter of 1 in. and were broken on a 6-in. span. The 0.875-in. and the 0.5-in. bars were broken at full diameters on a 6-in. span. In order to attempt a correlation between the 1-in. diameter and the substandard diameter test specimens, specimens were machined from the 2-in. diameter bars to 0.875-in. and 0.5-in. diameters. The results of the tests are given in Table XI. The values reported are the average of three individual tests in each case. Ordinary gray iron, cast in a standard arbitration bar 1.2 in. in diameter, will have impact values ranging from 20 to 40 ft-lb. if the iron is unalloyed. There are not sufficient data to set up a good relationship between impact and the diameters of the specimens, but preliminary calculations would indicate that the 0.875-in. and 0.5-in. diameter bars would have an equivalent impact resistance of about 30 ft-lb. if they are tested on larger specimens.

TABLE XI.—IMPACT TESTS ON PERMANENT MOLD IRONS.

Values are average of three individual tests.

Original Diameter, in.	Diameter Tested, in.	Span, in.	Impact, ft-lb.
2.........	1	6	18.8
1.2.......	1	6	22.0
0.875.....	0.875	6	15.7
0.5.......	0.5	6	8.0
2.........	0.875	6	12.7
2.........	0.5	6	4.1

Heat-Treated Properties:

Bars of 2-in., 1.2-in., 0.875-in., and 0.5-in. diameter were heated to 1525 F., held for 1 hr., and quenched in oil. They were then tempered at 850 F. for 1 hr. Tension specimens were machined from these bars. This procedure was followed in order to determine the physical properties of the irons in such a manner as to include the influence of mass during heat treatment. It did not seem logical, as is frequently done in tests of this type, to machine the test bars first and heat treat them subsequently, because the influence of mass on heat treatment would be lost. The results are given in Table XII.

TABLE XII.—TENSILE PROPERTIES, PLAIN AND HEAT TREATED.

Plain means fully annealed.

Heat-treated bars were quenched in oil from 1525 F and tempered 1 hr. at 850 F.

Original Diameter, in.	Treatment	Brinell Hardness	Tensile Strength, psi.	Modulus of Elasticity, psi.
2.0	Annealed	164	22 533	11 980 000
1.2		168	32 050	13 600 000
0.875		163	34 142	17 570 000
0.5		153	52 267	19 530 000
2.0	Heat Treated	206	32 242	10 950 000
1.2		258	38 783	13 360 000
0.875		310	50 250	17 400 000
0.5		330	82 433	19 200 000

The microstructures of these irons will be discussed later in the paper. These results are presented graphically in Fig. 4, where the diameter of the bar is plotted against the tensile strength. The tensile strengths of the annealed irons are included in this graph for purposes of comparison. It would appear from the results that, although high tensile properties may be obtained by heat treatment, these tensile values are obtained at a considerable sacrifice of softness and, therefore, machinability. It is interesting to note that the modulus of elasticity is not affected by heat treatment.

Growth Characteristics:

Gray cast irons are known to increase in volume or to "grow" when exposed to fluctuating high temperatures in various atmospheres. Specimens were machined 4 in. and $\frac{3}{8}$ in. in diameter and were placed in an electric muffle furnace. They were held at 1500 F. for $1\frac{1}{2}$ hr. The furnace atmosphere was still air. The current was turned off and the specimens were permitted to cool to considerably below the critical temperature, after which the heating cycle was repeated. After each 3 cycles the specimens were removed, the ends were given a light polish with emery paper to

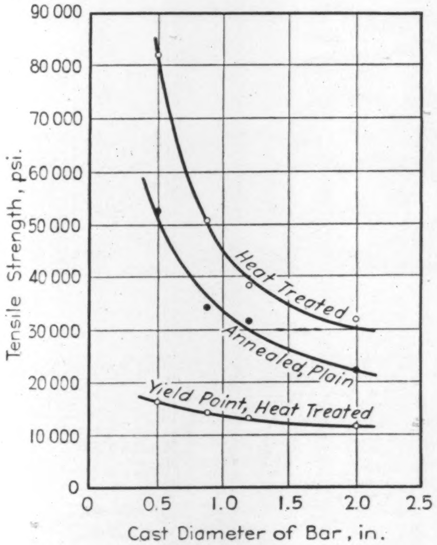


FIG. 4.—Influence Section of Size and Heat Treatment on Permanent Mold Iron.

TABLE XIII.—GROWTH OF PERMANENT MOLD IRONS.

Original Diameter of Iron, in.	Cycles	Increase in Length, per cent	Increase in Volume, per cent
2.0	3	0.198	0.595
	6	0.457	1.380
	9	0.783	2.360
	12	1.124	3.410
	15	1.442	4.390
	18	1.820	5.550
	21	2.260	6.920
	avg......	0.107	0.330
1.2	3	0.018	0.034
	6	0.180	0.540
	9	0.410	1.240
	12	0.658	1.985
	15	0.765	2.310
	18	0.992	3.008
	21	1.245	3.780
	avg......	0.060	0.180
0.875	3	0.098	0.294
	6	0.228	0.865
	9	0.472	1.420
	12	0.665	2.008
	15	0.804	2.430
	18	1.010	3.060
	21	.....	.....
	avg......	0.056	0.170
0.5	3	0.030	0.090
	6	0.176	0.528
	9	0.301	0.906
	12	0.449	1.350
	15	0.761	2.300
	18	0.830	2.510
	21	0.926	2.800
	avg......	0.044	0.133

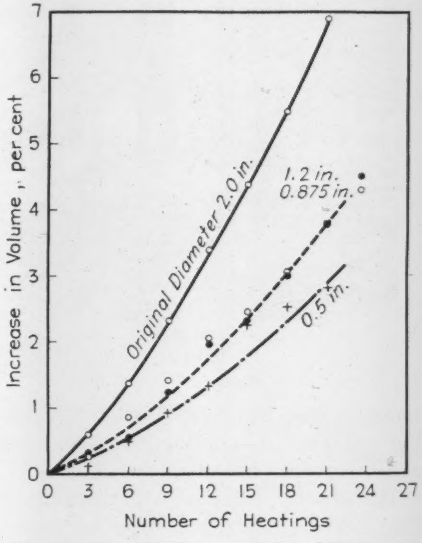
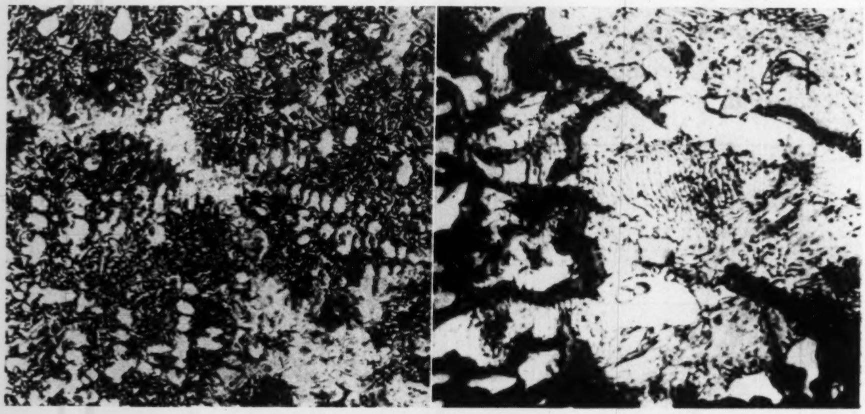


FIG. 5.—Growth Characteristics of Permanent Mold Iron.

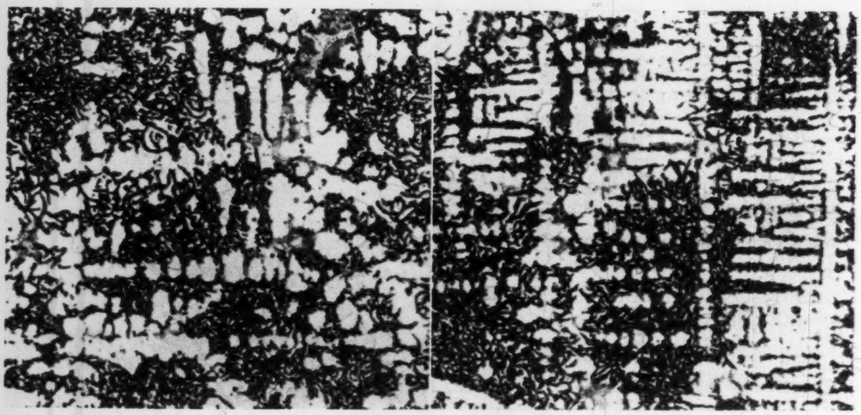
remove scale, and were measured with a micrometer device. A total of 21 cycles was employed in this test.

It was found that the specimens increased in length and the assumption was made that they must have increased in diameter by a proportionate amount. The increase in volume was computed and the values are given in Table XIII.



× 100. × 1000.

FIG. 6.—As Cast, Etched.



Center. Outside.

FIG. 7.—0.5-in. Bar Annealed, Etched (× 100).

The results are shown graphically in Fig. 5 where the percentage increase in volume is plotted against the number of heating cycles. From these values it may be concluded that the larger the bar the greater the amount of growth. If these values for growth are compared with figures published by other investigators,[5] no direct relationships may be derived. This is true because in the various tests presented no two investigators used the same heating cycle with respect to time or temperature, nor did they use the same atmospheres. These values herein presented are merely com-

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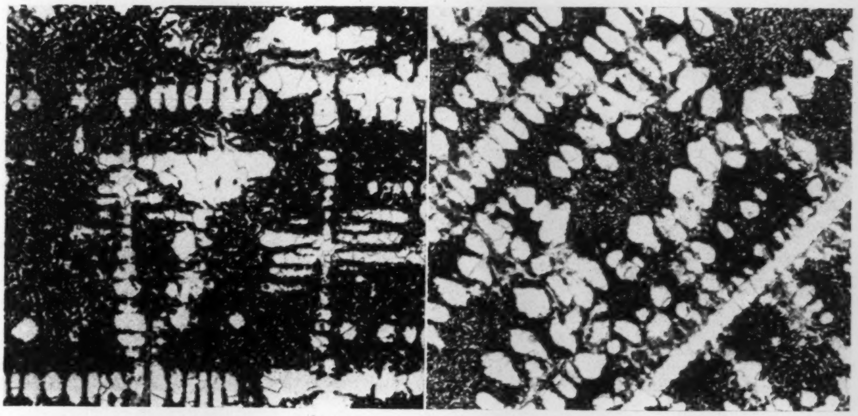
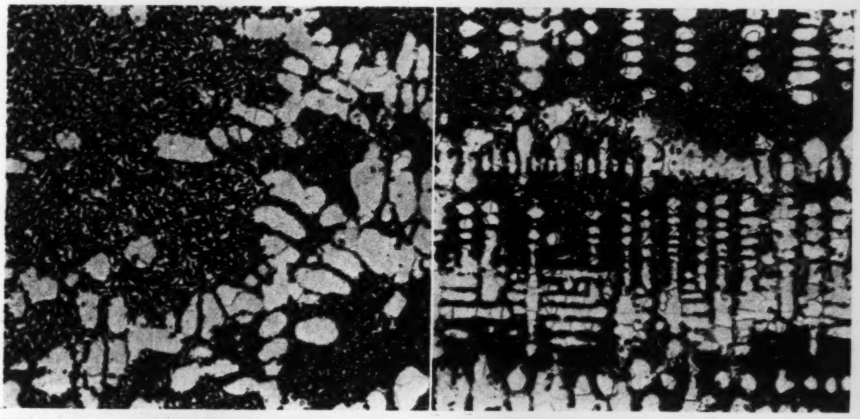
[5] "Alloy Cast Irons," p. 106, Am. Foundrymen's Assn. (1939).



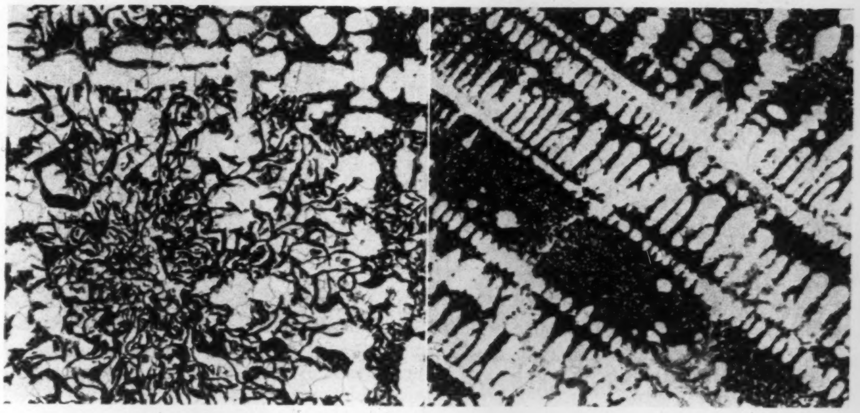
Center. Outside.

FIG. 8.—0.875-in. Bar Annealed, Etched (× 100).



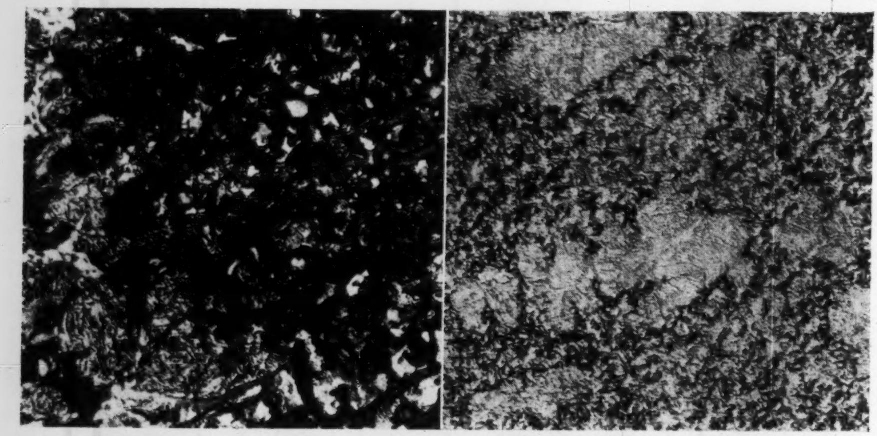
Center. Outside.

FIG. 9.—1.2-in. Bar Annealed, Etched (× 100).



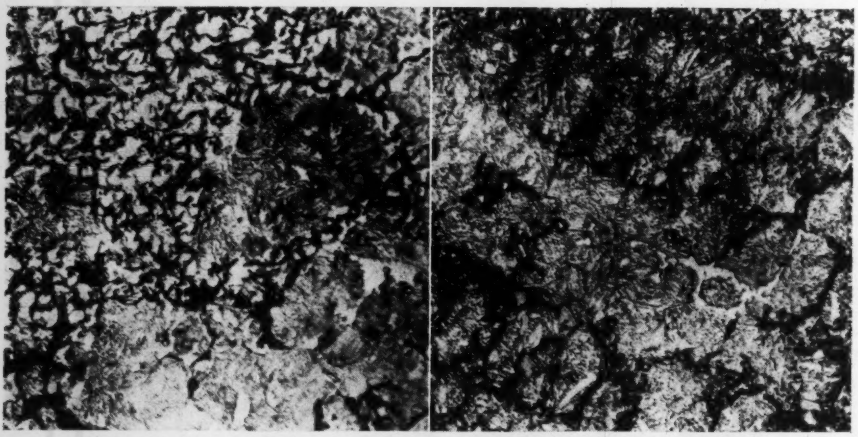
Center. Outside.

FIG. 10.—2.0-in. Bar Annealed, Etched (× 100).



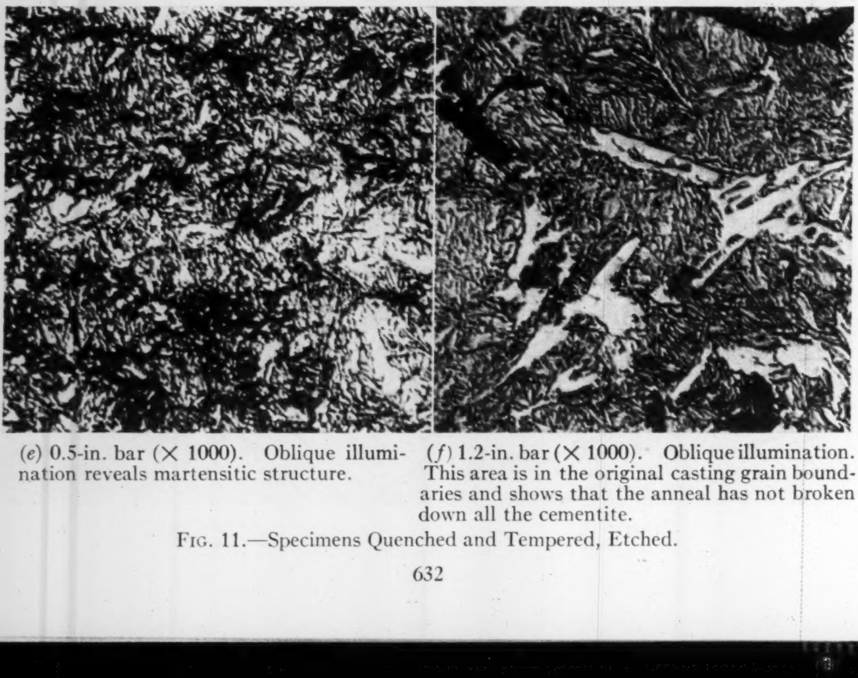
(*a*) 0.5-in. bar (× 500).

(*b*) 0.875-in. bar (× 500).



(*c*) 1.2-in. bar (× 500).

(*d*) 2.0-in. bar (× 500).



(*e*) 0.5-in. bar (× 1000). Oblique illumination reveals martensitic structure.

(*f*) 1.2-in. bar (× 1000). Oblique illumination. This area is in the original casting grain boundaries and shows that the anneal has not broken down all the cementite.

Fig. 11.—Specimens Quenched and Tempered, Etched.

parative between section sizes of permanent mold iron for the conditions used in this particular test.

Microstructure:

Specimens were prepared for microstructure examination from specimens of each size bar in the as-cast condition. Figure 6 shows typical structures at 100 × and 1000 ×. It is readily seen that the matrix consists of ferrite with some pearlite. A considerable amount of phosphide eutectic may be seen at the austenite grain boundaries. The graphite is of fine eutectiform quality.

Specimens were likewise prepared from samples of annealed bars, that is, the condition in which the castings are sold. Typical structures are shown in Figs. 7 to 10, which present at 100 × typical sections from the outside and center of each size bar.

As may be expected, the graphite is finer and the ferrite cell size is smaller at the outside side of the bar than at the center, and the ferrite and graphite are finer in a smaller bar than they are in a larger bar. There is no great qualitative difference in the microstructures of any of these irons except that shown in the center of a 2-in. bar. Here the graphite has assumed a small flake form rather than the eutectiform appearance shown by all the others. This flake graphite is typical of ordinary gray cast irons and should, in a measure at least, explain the lower physical properties of the larger bars.

Specimens were also prepared from each size bar in the heat-treated condition. The nature of heat treatment was described in the section of this paper entitled "Heat-Treated Properties." The microstructures are shown in Fig. 11. It may be seen that the matrix was decomposed martensite.

CORRELATION BETWEEN PROPERTIES AND SECTION SIZE

In ordinary sand-cast gray cast irons the section size directly controls the rate of cooling of the iron from the molten state to room temperature. It has been found that the rate of cooling profoundly influences the metallurgical and mechanical properties of the product.

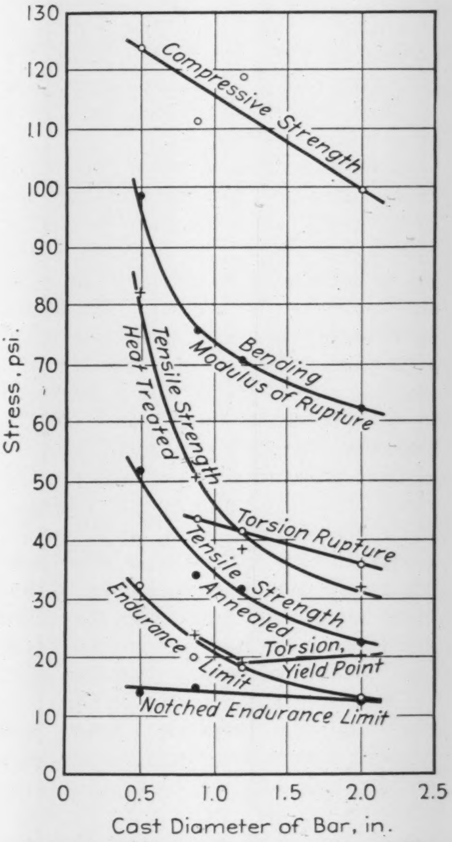


FIG. 12.—Influence of Section Size on Mechanical Properties.

In a series of irons cast from the same ladle of molten iron, the castings having small section size, a rapid rate of cooling, will possess a higher strength, in tension and other ways, than castings of heavier section size and slower rates of cooling. The difference in properties is also reflected to a large extent in the microstructure. In rapidly cooled sections the graphite flakes will be small and the pearlite of a fine lamellar nature. As the section size increases, the size of the graphite flakes and the pearlite lamellar gets larger. With even slower rates of cooling in the large section sizes, the microstructure will consist of very coarse graphite flakes, coarse pearlite and a large amount of ferrite.

In permanent mold irons, due to the rapid extraction of heat through the metal mold, the difference in cooling rate between a ½-in. bar and 2-in. bar is not so great as it would be if the same size bars were cast in sand. The microstructures in the as-cast conditions reveal a progressive increase in crystal size and graphite size as the section is increased in the permanent mold irons. In the 2-in. size, there is a very small quantity of flake graphite near the center. The matrix structure before and after annealing is practically identical for all section sizes. In view of these facts, it is somewhat surprising that such large differences in mechanical properties are found.

Figure 12 has been prepared to show the influence of section diameter upon the mechanical properties. In this chart the following properties have been plotted: compressive strength, modulus of rupture in bending, torsional rupture strength, tensile strength in the heat-treated condition, tensile strength in the annealed condition, torsional yield point, endurance limit, and notched endurance limit.

The values for all of these different methods of testing are higher in the case of small bars than larger bars. The yield point in torsion and the notched endurance limit apparently are independent of section size.

Figure 13 has been prepared to show the influence of section diameter on the moduli in bending, tension, and torsion. In all cases the irons are stiffer in smaller diameters than in heavier diameters. In referring back to Table IV, it may be seen that the Brinell hardness does not vary appreciably with section diameter although it is surprising to note that the smallest bar exhibits the lowest hardness.

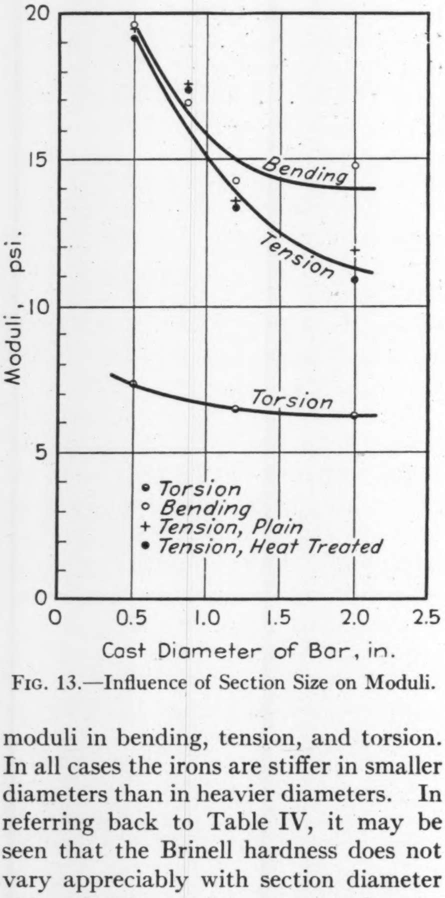


FIG. 13.—Influence of Section Size on Moduli.

DISCUSSION

Mr. C. W. MacGregor.[1]—Table I of the paper gives values of the tensile strength and the modulus of rupture in shear for permanent mold castings. It will be noted that these values differ greatly in magnitude. This is no doubt caused by the fact that the elementary elastic formulas for shearing stress were used to compute the shearing stress at rupture. Actually, of course, these formulas hold only below the elastic limit.

As soon as the elastic limit in shear is exceeded, the shearing stresses begin to redistribute themselves over the cross-sections of the twisted bar. For a torque moment well above that necessary to initiate yielding, the shearing stresses no longer follow a linear distribution as in the elastic case, but are distributed somewhat parabolically over the cross-section.

The actual shearing stress in the outside layer of a round bar under torsion can be calculated accurately at any torque moment M_t and for an arbitrary torque-twist curve from the formula

$$S_a = \frac{1}{2\pi a^3}\left[3M_t + \theta\,\frac{dM_t}{d\theta}\right] \ldots (1)$$

where S_a, a, M_t, and θ represent the shearing stress in the outside fiber, the radius of the bar, the torque moment and the unit angle of twist, respectively. This of course means that the torque twist curve must be determined from experiment which can be readily carried out.

Equation 1 also has a very simple graphical interpretation which is the way in which it is used practically. For a discussion of this method reference is made to the work of Nadai.[2]

This method has been applied by the writer to torsion tests on cast iron[3] in which it was found that the actual shearing stress present in the bar at fracture agreed with the tensile strength to within 3 per cent. For an ideally brittle material these quantities should be exactly equal to each other.

Mr. Richard Schneidewind[4].—I have looked over Mr. MacGregor's Eq. 1, which is Eq. 10 of Nadai's book on "Plasticity," and I follow the derivation. I checked the equation against the perfectly elastic case as follows:

given $\theta = \dfrac{M_t}{GI_p}$ then $\theta\,\dfrac{dM}{d\theta} = \dfrac{M_t}{GI_p}\cdot GI_p = M_t$. Accordingly

$$S_a = \frac{1}{2\pi a^3}\left(\theta\,\frac{dM}{d\theta} + 3M_t\right)$$

$$= \frac{1}{2\pi a^3}\,(M_t + 3M_t) = \frac{2M_t}{\pi a^3}$$

This checks the usual formula $= \dfrac{16M_t}{\pi d^3}$.

Now if an (M_t, θ) curve (see accompanying Fig. 1) is assumed such as we know exists for cast iron, say as an extreme case let $\frac{dM}{d\theta} = \theta$ at the end point, then S_a has a minimum value of

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[1] Professor of Applied Mechanics, Massachusetts Institute of Technology, Cambridge, Mass.

[2] A. Nadai, "Plasticity," McGraw-Hill Book Co., p. 128, New York, N. Y. (1931).

[3] C. W. MacGregor and J. A. Hrones, "Recent Investigations in Plastic Torsion," *Journal of Applied Mechanics*, December, 1937, pp. A163–A169.

[4] Associate Professor of Metallurgical Engineering, Department of Chemical and Metallurgical Engineering, University of Michigan, Ann Arbor, Mich.

$$S_a = \frac{1}{2\pi a^3}\left(\theta\frac{dM}{d\theta} + 3M_t\right)$$

$$= \frac{1}{2\pi a^3}(0 + 3M_t) = \frac{3M_t}{2\pi a^3}$$

which is three-fourths of that for the elastic case.

From the foregoing discussion, it would appear that your true S_a must lie somewhere between the modulus of rupture in torsion and three-fourths of that value.

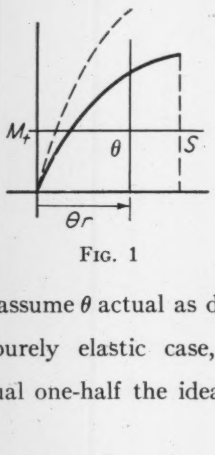

FIG. 1

Say we assume θ actual as double that for the purely elastic case, and $\frac{dM}{d\theta}$ actual equal one-half the ideal, then S_a would be

$$S_a = \frac{1}{2\pi a^3}\left(2\cdot\frac{M_t}{2} + 3M_t\right)$$

$$= \frac{2M_t}{\pi a^3} = \frac{16M_t}{\pi d^3}$$

which is identically the modulus of rupture.

For the stress-strain curve of cast iron, it is evident that the S_a of the formula mentioned by Mr. MacGregor gives values very close to the values of the modulus of rupture in torsion. Proof that the modulus of rupture is never less than that given in the formula follows:

For a solid circular shaft

$$M_t = \int_0^a 2\pi r dr \cdot f(r\theta)\cdot r$$

for a given S_a the stress at radius r which is $f\ (r\theta)$ which is BA (accompanying Fig. 2) is always greater than for the elastic case (CB).

In obtaining our values we assumed the elastic case, which is undoubtedly wrong for cast irons. We do not know the degree of plasticity, and so the values should lie between the ones reported and the values for tensile strength.

MR. J. M. LESSELLS.[5]—The above paper adds quite useful data to those already available on permanent mold irons.

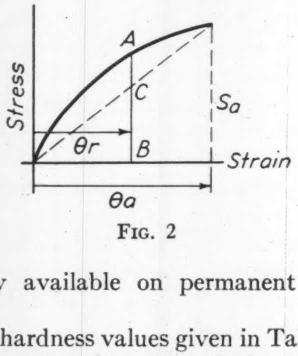

FIG. 2

The hardness values given in Table IV of the paper show no relation to the tensile strength as given in Table II. The hardness is constant for the 2.0- , 1.2- , and 0.875-in. diameters, while the tensile strengths vary from 22,533 psi. to 34,142 psi., respectively. Can we conclude that this is a residual stress effect? As regards fatigue, Peterson[6] has shown that there is little, if any, size effect with cast iron. In the case of permanent mold iron the present paper shows there is a considerable effect due to the size of the original cast bar. The very low difference found for fatigue under stress concentration seems to agree with other published results.

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[5] Associate Professor of Mechanical Engineering, Engineering Dept., Massachusetts Institute of Technology, Cambridge, Mass.

[6] R. E. Peterson, "Stress Concentration Phenomena in Fatigue of Metals," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 55, p. 157 (1933).

If the authors would advise as to location of the fatigue test bars in the section of the original bar this would be helpful in further studies.

MR. SCHNEIDEWIND.—Our work was not that refined that we made those measurements, but this material was fully annealed so we assumed the stresses would at least be low. Of course, they were not drawn after heat treatment, so there might be some machining stresses in there, but the material was fully annealed.

Transverse bars were broken. Fatigue specimens were made from the center sections near the transverse fracture.

I do not believe that residual stresses can account for all the differences in tensile strength. The size, shape, and distribution of the graphite phase and differences in grain size of the ferrite undoubtedly contribute.

In malleable iron the process of graphitization accomplished is accompanied by some expansion of graphite. I believe some residual stresses in the metal surrounding graphite are high. On prolonged heating of that type of iron, we got germination and enormous grain growth around graphite particles.

MR. J. S. VANICK.[7]—This information on the physical properties of permanent mold cast iron is so rare that I was moved to make a little contribution here from some data that we had in storage.

The test bars that were used were 2-in. diameter chill cast or permanent mold cast and had been annealed, and tested in accordance with the requirements for test bar C of A.S.T.M. Specifications A 48.[8] The values for unalloyed compositions similar to Mr. Schneidewind's were very similar to his values, and when alloyed with a nickel-chromium - molybdenum composition they ran about 10,000 psi. higher after annealing—those were cast gray. When the silicon content dropped to about 1.10 per cent and they came out white and were annealed to a sort of malleable, they picked up another 10,000 psi. so they were running up around 53,000 psi. tensile strength—in a 2-in. bar with a structure composed of temper carbon—in a pearlitic or martensitic matrix. (See accompanying Table I.)

TABLE I.—COMPARISON OF SAND-CAST *versus* ALLOYED ANNEALED CHILL-CAST IRONS, 2-IN. DIAMETER BARS.

	Analysis, per cent								Brinell Hardness Number	Tensile Strength, psi., A.S.T.M. Type "C" Bar
	Total Carbon	Silicon	Manganese	Sulfur	Phosphorus	Nickel	Chromium	Molybdenum		
Sand-cast	3.56	1.58	0.47	0.10	0.60	....	....	....	199	26 300
Sand-cast	3.65	1.24	0.53	0.10	0.11	....	....	....	183	29 500
Chill-cast	3.59	2.22	0.65	0.09	0.21	0.26	0.06	0.02	175	30 100
Chill-cast	3.46	2.23	0.64	0.08	0.20	0.92	0.36	0.69	209	42 500
Chill-cast and annealed	3.64	1.15	0.70	0.06	0.18	0.92	0.36	0.72	243	53 000

These values were for permanent mold cast irons and are particularly interesting if you realize, as Mr. Schneidewind pointed out, that sand-cast bars in these high-carbon compositions may run as low as 18,000 to 22,000 psi. tensile strength. In these big sections they can be brought up to 30,000 psi. in the permanent mold casting—40,000 psi. when alloyed and 50,000 psi. when alloyed and put through the tempering anneal.

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[7] Metallurgist, The International Nickel Co., Inc., New York, N. Y.

[8] Standard Specifications for Gray Iron Castings (A 48 - 41), 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 95.

I should like to ask Mr. Schneidewind whether he has done any work on inoculating these irons, he being an expert at that.

MR. SCHNEIDEWIND.—I, personally, have not done any work on that but I know that Max Kuniansky, of the Lynchburg Foundry Co., who makes permanent mold pipe, has. He is inoculating irons of this general type.

MR. H. A. SCHWARTZ.[9]—I may be able to throw some light on Mr. Schneidewind's suggestion that there might be stresses accompanying graphitization.

A year or two ago in some informal things we were doing with Mr. Wells at the Carnegie Institute of Technology we offered to anneal some of his very high purity alloys for a very long time—six months. He made those materials—they contained about 0.75 per cent combined carbon—and sealed them in a quartz tube in argon at about a third of an atmosphere pressure, so when they were heated to just below the critical point the argon was approximately at room pressure. We heated two of those capsules for this six-month period at a temperature of a little under 1400 F. When the samples were examined after they came out, one of the tubes, unfortunately, broke, but the other tube which was intact contained a lot of gray flakes that were not there when the tube was sealed. We did not feel at liberty to break the tube before we sent the material back to Carnegie Institute, but it turned out that these specimens were now something like 0.35 per cent total carbon, a drop of about 0.4 per cent. The carbon had not deposited inside the metal in that length of time but had gone clear out of the specimen and crystallized. The only reason I can think of for that is the reduction of free energy which the system would suffer if the graphite were removed and went clear to the outside rather than to remain inside under the mechanical forces of expansion.

I will trade that statement for a question. I should like to ask Mr. Schneidewind, what is the yield point of gray iron in tension? How is that defined?

MR. SCHNEIDEWIND.—That particular set of tension tests was conducted by our Engineering Mechanics Dept., which drew stress-strain curves. They put on their best instruments, and at the deviation from a straight line they labeled that yield point.

MR. SCHWARTZ.—Mr. Moore, is that yield point proper?

MR. H. F. MOORE.[10]—It is not.

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[9] Manager of Research, National Malleable and Steel Castings Co., Cleveland, Ohio.

[10] Professor of Engineering Materials, University of Illinois, Urbana, Ill.

THE NOTCH SENSITIVITY OF ALLOYED CAST IRONS SUBJECTED TO REPEATED AND STATIC LOADS

By W. Leighton Collins[1] and James O. Smith[1]

Synopsis

Four cast irons of class 20, 30, 40, and 50,[2] respectively, and ranging in chemical composition from an ordinary gray iron to highly alloyed irons were investigated for their sensitivity to the effect of mechanical and corrosion notches under repeated and static loads at room temperatures. For repeated bending, torsional, and axial loading, respectively, it was found that ordinary gray cast iron is relatively insensitive to the effects of a notch, but that as the static tensile strength of the irons increases the notch sensitivity to repeated loading increases; however this tendency is less pronounced for the class 40 (innoculated) iron tested than for the other irons tested. Contrary to the general impression, the four cast irons investigated were found to be insensitive to the effects of a notch under static tensile, compressive, and torsional loads, respectively.

The general impression regarding the notch sensitivity of cast iron seems to be that it is relatively insensitive to the effect of notches when subjected to repeated loads, and that on the other hand, it *is* very sensitive to the effect of notches when subjected to static loads. Notch sensitivity as here used means the ratio of the strength of an unnotched specimen to the strength of a notched specimen. The widespread development and use of a great variety of cast irons, many of them designed for special uses requiring high strength, such as cast crankshafts, or for use to resist heat, corrosion, wear, etc., gives rise to the question as to how sensitive these irons are to the effect of notches when subjected to repeated or to static loads.

To provide information to answer this question for a variety of irons, (information is available for a variety of steels[3, 4]) four cast irons whose analyses are given in Table I were used for this investigation. Repeated load tests were made for completely reversed cycles of stress due to bending, torsional and axial loads, respectively. Both unnotched and notched specimens were used for each of the three types of loading just mentioned. The notch in the bending and torsional specimens consisted of a small transverse hole and in the axial specimens the notch was a circumferen-

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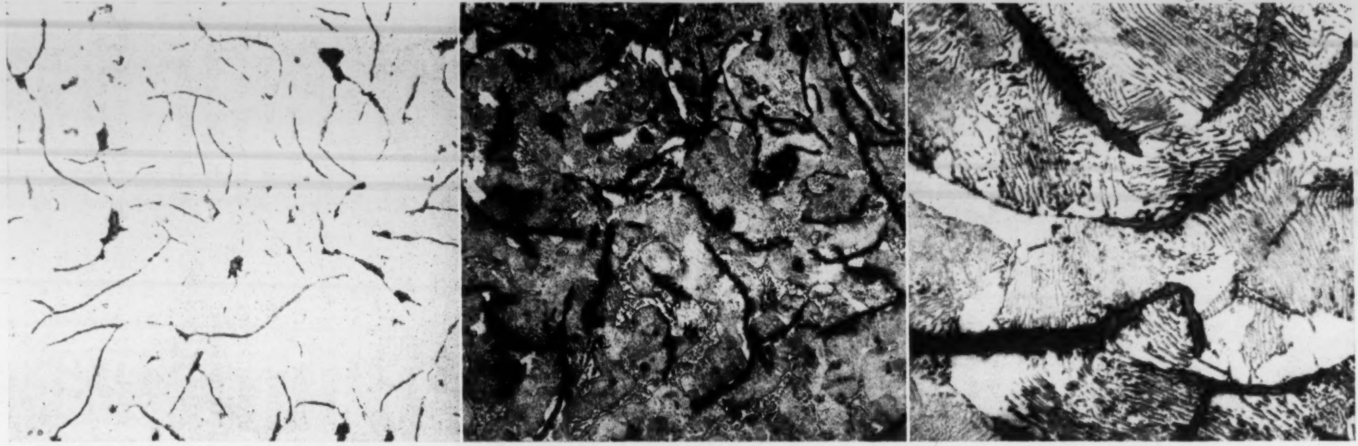
[1] Assistant Professor, and Associate, respectively, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[2] Standard Specifications for Gray-Iron Castings (A 48 – 36), 1939 Book of A.S.T.M. Standards, Part I, p. 482.

[3] T. J. Dolan, "Simultaneous Effects of Corrosion and Abrupt Changes in Section on the Fatigue Strength of Steel," *Journal of Applied Mechanics*, Vol. 5, December, 1938, P.A., p. 141.

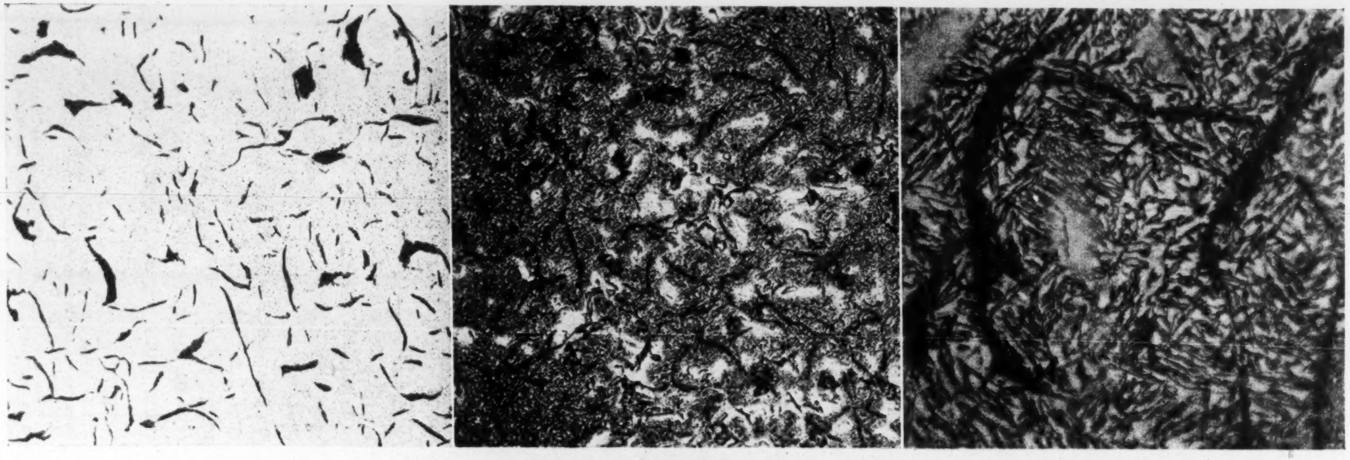
[4] W. Leighton Collins and T. J. Dolan, "Physical Properties of Four Low-Alloy High-Strength Steels," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part II, p. 157 (1938).

Unetched (× 100). Etched in 2 per cent Nital (× 100). Etched in 2 per cent Nital (× 500).



(*a*) Iron A.

Specimens taken from mid-length of casting at center of one-quarter of cross-section.

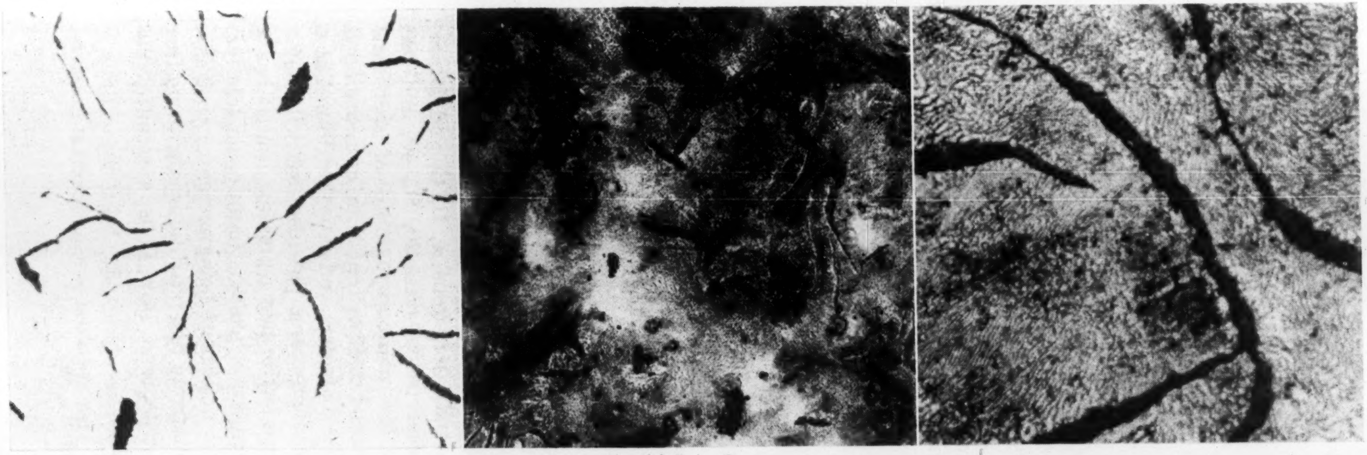


(*b*) Iron B.

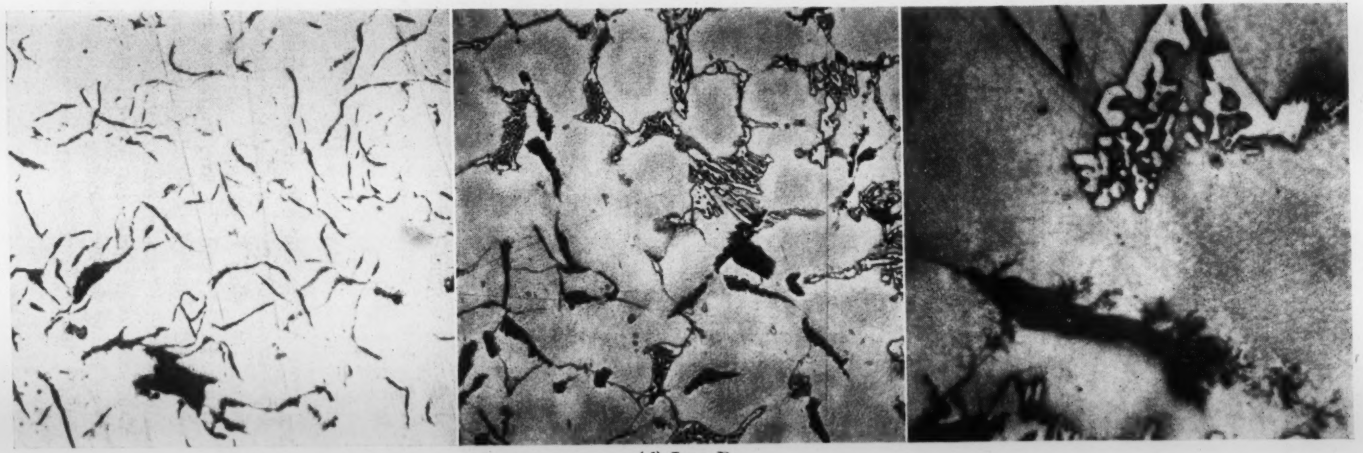
NT IN BINDING

(*b*) Iron B.

Unetched (× 100). Etched in 2 per cent Nital (× 100). Etched in 2 per cent Nital (× 500).



(*c*) Iron C.
Specimens taken from one-quarter point of thickness near center of casting.



(*d*) Iron D.
Specimens taken from one-quarter point of thickness near center of casting.

FIG. 1.—Typical Photomicrographs.

tial V-groove. In bending and torsion each type of specimen was also tested under a stream of fresh tap water whose pH factor is about 7.3. Static load tests of unnotched and notched specimens were also made in tension, compression, and torsion, respectively. The notch in the static load test specimens consisted of a circumferential V-groove.

This program of tests to investigate the notch sensitivity of cast iron seems adequate since the failure of cast iron due to the action of loads is nearly always by brittle fracture. Hence such factors as a triaxial state of stress, low temperature, high speed of straining, effect of size, etc., which have a tendency to render some ductile metals quite sensitive to the effects of a notch, probably have little or no effect in rendering cast irons more sensitive to notch effects than was found for the tests just outlined and therefore are not considered in this report.

TABLE I.—CHEMICAL COMPOSITIONS.

Material	Total Carbon, per cent	Free Carbon, per cent	Silicon, per cent	Manganese, per cent	Sulfur, per cent		Phosphorus, per cent	Nickel, per cent	Chromium, per cent	Molybdenum, per cent	Copper, per cent	Titanium, per cent	Vanadium, per cent	Arsenic, per cent
					Evolved as H_2S	Gravimetric								
A	3.32	2.56	1.75	0.51	0.127	0.138	0.48	0.04	0.01	0.00	0.05	....	.....	.....
B	3.11	2.30	2.23	0.66	0.075	0.104	0.10	1.49	0.09	0.79	0.18	0.025	0.010	0.021
C	2.84	2.10	1.52	1.05	0.106	0.124	0.07	0.20	0.31	0.00	0.37	0.020	0.005	0.013
D	2.63	1.96	2.14	1.23	0.059	0.065	0.16	14.90	2.09	0.00	6.94	0.022	0.005	0.012

MATERIALS

Iron A is a slightly alloyed ordinary gray iron cast in the Machine Laboratories of the University of Illinois. Irons B, C, and D are commercially produced irons of three different kinds and were all made under normal production conditions, no special precautions being made to secure an unusually sound iron. Iron B can be classified as a high-strength nickel-molybdenum iron, iron C as a slightly alloyed nickel-chromium-copper innoculated iron, and iron D as a high-nickel-copper-chromium iron of the austenitic type.

The typical microstructure of each iron is shown in Fig. 1. The specimens for the photomicrographs came from the same location in the thickness of the castings as the centers of the test specimens. A comparison of the unetched photomicrographs with the A.S.T.M.-A.F.A. Charts for Evaluating the Microstructure of Graphite in Gray Iron[5] indicates that all irons are of type A, random orientation of graphite flakes, and that, in general, the graphite flakes are size 4, 0.5 to 1.0 in. in length. Irons A and D have some flakes over 1 in. in length; consequently, for those irons some flakes are also size 5. As will be noted later, irons A, B, C, and D are, respectively, class 20, 50, 40, and 30.[2]

The photomicrographs of specimens etched in 2 per cent Nital and magnified 500 diameters, indicate that iron A is a medium-coarse pearlitic iron with some free ferrite being present; iron B has a feathery acicular structure, no free ferrite or pearlite being present; iron C is a relatively fine pearlitic iron with no free ferrite, and iron D is austenitic, the

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[5] 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 300.

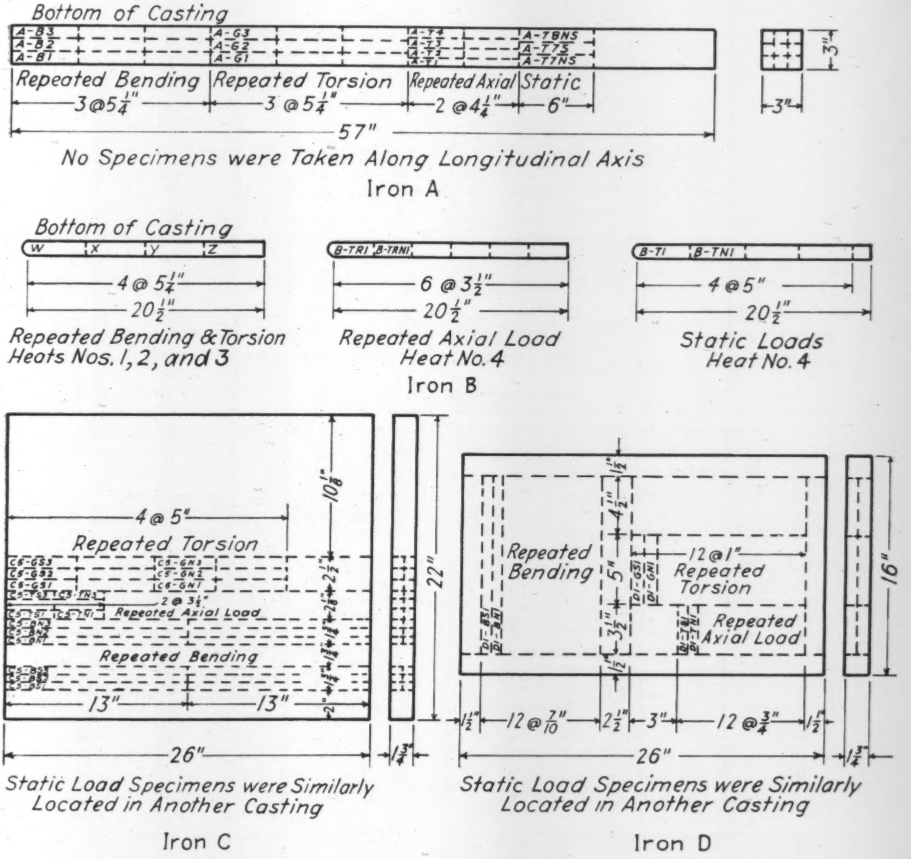

Bottom of Casting
A-B3
A-B2
A-B1
A-G3
A-G2
A-G1
A-T4
A-T3
A-T2
A-T1
A-T8NS
A-T7S
A-T7NS
Repeated Bending
Repeated Torsion
Repeated Axial
Static
3 @ 5¼"
3 @ 5¼"
2 @ 4¼"
6"
57"
3"
3"
No Specimens were Taken Along Longitudinal Axis
Iron A
Bottom of Casting
W
X
Y
Z
4 @ 5¼"
20½"
Repeated Bending & Torsion
Heats Nos. 1, 2, and 3
B-TR1
B-TRN1
6 @ 3½"
20½"
Repeated Axial Load
Heat No. 4
B-T1
B-TN1
4 @ 5"
20½"
Static Loads
Heat No. 4
Iron B
4 @ 5"
Repeated Torsion
C5-GS3
C5-GS2
C5-GS1
C5-GN3
C5-GN2
C5-GN1
C5-TS
C5-TN
2 @ 3½"
C5-TS1
C5-TN1
Repeated Axial Load
Repeated Bending
10⅛"
2¼"
22"
13"
13"
26"
1¾"
Static Load Specimens were Similarly Located in Another Casting
Iron C
Repeated Bending
12 @ 1"
Repeated Torsion
Repeated Axial Load
1½"
4½"
5"
3½"
16"
1½"
12 @ 7/10"
2½"
3"
12 @ ¾"
1½"
26"
1¾"
Static Load Specimens were Similarly Located in Another Casting
Iron D

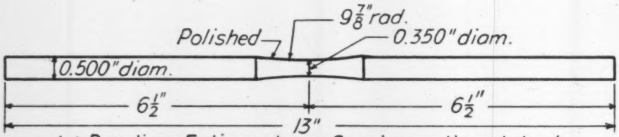
FIG. 2.—Typical Location and Marking of Specimens.

graphite being at the austenite grain boundaries and the irregularly shaped areas apparently being of eutectic composition.

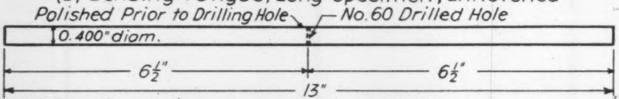
Iron A was a casting 3 by 3 in. in cross-section and 56 in. long, cast with the 56-in. dimension vertical. Iron B was in the form of transverse test bars 1.2 in. in diameter and 20.5 in. long; bars from four heats of the same type of iron were used, the heat analyses being as follows:

Heat	Total Carbon, per cent	Manganese, per cent	Sulfur, per cent	Phosphorus, per cent	Silicon, per cent	Nickel, per cent	Chromium, per cent	Molybdenum, per cent
No. 1......	3.05	0.79	0.116	0.163	2.44	1.48	0.09	0.91
No. 2......	3.14	0.65	0.135	0.166	2.20	1.59	0.08	0.92
No. 3......	3.22	0.79	0.111	0.175	2.09	1.79	0.03	0.91
No. 4......	3.16	0.69	0.100	0.149	2.11	1.56	0.10	0.82

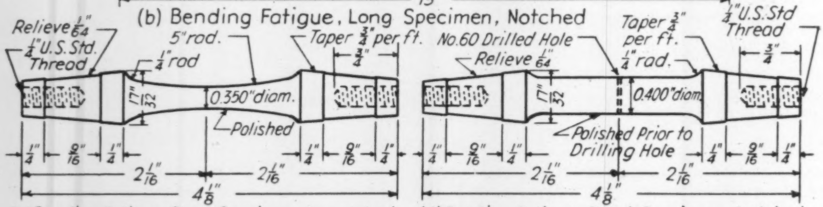
Irons C and D were in the form of vertically cast plates 1¾ in. thick, 26 in. long, and 22 in. and 16 in. high, respectively. All castings for irons A, C, and D were poured from one ladle.


9 7/8" rad.
Polished
0.350" diam.
0.500" diam.
6 1/2"
6 1/2"
13"

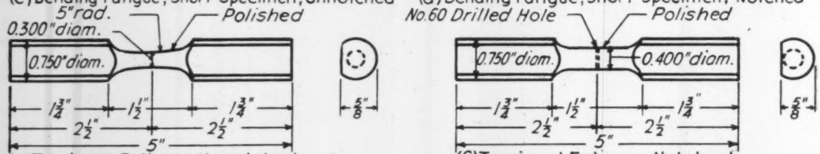
(a) Bending Fatigue, Long Specimen, Unnotched


Polished Prior to Drilling Hole
No. 60 Drilled Hole
0.400" diam.
6 1/2"
6 1/2"
13"

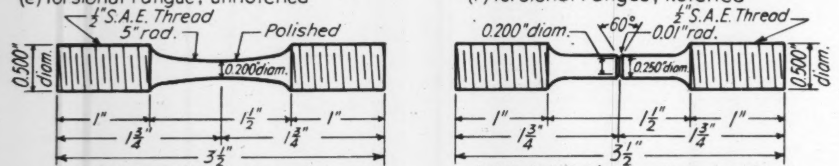
(b) Bending Fatigue, Long Specimen, Notched


Relieve 1/64"
1/4" U.S. Std. Thread
5" rad.
1/4" rad
Taper 3/4" per ft.
3/4"
17/32"
0.350" diam.
Polished
1/4"
9/16"
1/4"
2 1/16"
2 1/16"
4 1/8"
No. 60 Drilled Hole
Relieve 1/64"
Taper 3/4" per ft.
1/4" rad.
1/4" U.S. Std Thread
0.400" diam.
Polished Prior to Drilling Hole

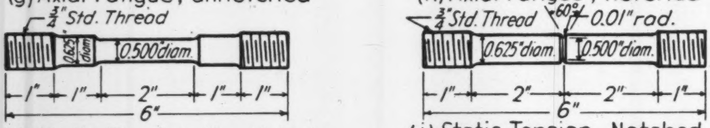
(c) Bending Fatigue, Short Specimen, Unnotched (d) Bending Fatigue, Short Specimen, Notched


5" rad.
Polished
0.300" diam.
0.750" diam.
1 3/4"
1 1/2"
1 3/4"
2 1/2"
2 1/2"
5"
5/8"
No. 60 Drilled Hole
Polished
0.750" diam.
0.400" diam.

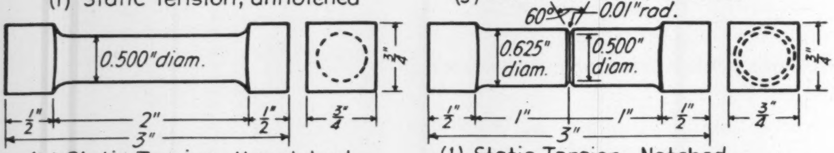
(e) Torsional Fatigue, Unnotched (f) Torsional Fatigue, Notched


1/2" S.A.E. Thread
5" rad.
Polished
0.500" diam.
0.200" diam.
1"
1 1/2"
1"
1 3/4"
1 3/4"
3 1/2"
0.200" diam.
60°
0.01" rad.
1/2" S.A.E. Thread
0.250" diam.

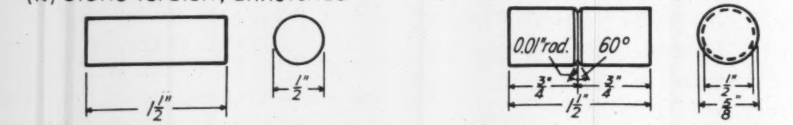
(g) Axial Fatigue, Unnotched (h) Axial Fatigue, Notched


3/4" Std. Thread
0.625" diam.
0.500" diam.
1"
1"
2"
1"
1"
6"
60°
0.01" rad.
0.625" diam.
0.500" diam.
2"
2"

(i) Static Tension, Unnotched (j) Static Tension, Notched


0.500" diam.
1/2"
2"
1/2"
3"
3/4"
3/4"
60°
0.01" rad.
0.625" diam.
0.500" diam.
1/2"
1"
1"
1/2"
3"

(k) Static Torsion, Unnotched (l) Static Torsion, Notched


1 1/2"
1/2"
0.01" rad.
60°
3/4"
3/4"
1 1/2"
1/2"
5/8"

(m) Static Compression, Unnotched (n) Static Compression, Notched

FIG. 3.—Types of Specimens.

All fatigue specimens were polished longitudinally with No. 00 emery cloth.
All static load specimens had tool marks removed with a fine file.

TEST SPECIMENS

Typical locations of specimens for each iron are shown in Fig. 2. It will be noted that a record of the location of each specimen in the casting was maintained. The code for marking specimens usually was such that the type of iron, casting number, type of specimen, type of load, etc., could be determined readily.

The various kinds of specimens used are shown in Fig. 3. The short specimens were used in bending fatigue when the quantity of material available was limited. In all other cases the same kinds and sizes of specimens were used

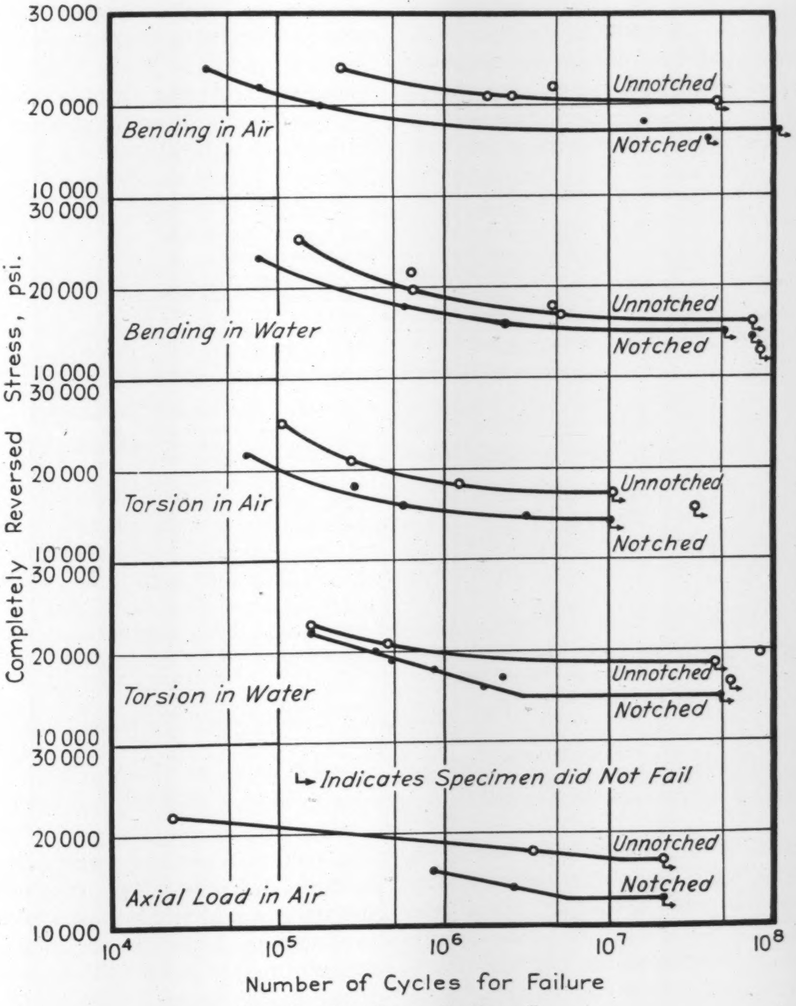


FIG. 4.—*S–N* Curves for Iron C.
Completely reversed cycles of stress.

for all irons. For the repeated loads in bending and torsion all unnotched specimens had a minimum diameter of 0.350 in., the notched specimens had a diameter of 0.400 in., and the notch consisted of a small transverse hole whose diameter was 0.10 the diameter of the specimen. For repeated axial loads the minimum diameter was 0.200 in., the size being governed by the capacity of the machines; the notch was a circumferential 60-deg. V-groove having a depth of 0.025 in. and a radius at the root of the notch of 0.01 in. In all the static load tests, whether tension, compression, or torsion, the nominal diameter at the root of the notch was the same as the nominal diameter of the unnotched specimens and was always approximately equal to 0.500 in. The notch in all the static load test specimens consisted of a circumferential 60-deg. V-groove 0.05 in. deep and having a radius of 0.01 in. at the root.

TABLE II.—ENDURANCE LIMITS AND STRESS CONCENTRATION FACTORS FOR COMPLETELY REVERSED CYCLES OF STRESS.

$$K = \frac{\text{Endurance Limit of Unnotched Specimen Tested in Air}}{\text{Endurance Limit of Notched Specimen Tested in Air}}$$

$$K_c = \frac{\text{Endurance Limit of Specimen Tested in Air}}{\text{Endurance Limit of Same Type of Specimen Tested in Water}}$$

$$\text{Total Stress-Concentration Factor} = K \times K_c$$

Iron	Type of Specimen	Bending				Torsion				Axial Load		Static Tensile Strength, psi.
		Endurance Limit, psi.		Stress-Concentration Factor (See Note)		Endurance Limit, psi.		Stress-Concentration Factor (See Note)		Endurance Limit, psi.	Stress-Concentration Factor (See Note)	
		In Air	In Water	K	K_c	In Air	In Water	K	K_c	In Air	K	
A.......	Unnotched	9 000	7 000	....	1.29	11 000	7 900	....	1.25	6 500	....	22 200
	Notched	8 000	5 000	1.12	1.60	8 000	8 100	1.37	0.99	6 000	1.08	
B.......	Unnotched	21 000	8 000[a]	....	2.62	22 000	12 000[a]	....	1.83	19 800	....	53 400
	Notched	14 000	5 000	1.50	2.80	16 000	8 000[b]	1.38	2.00	16 400	1.21	
C.......	Unnotched	20 100	16 000	....	1.26	17 100	18 500[c]	....	0.92	17 000	....	46 000
	Notched	17 000	15 000	1.18	1.13	14 300	15 000	1.20	0.95	13 000	1.31	
D.......	Unnotched	12 000	6 000	....	2.00	12 000	6 000[a]	....	2.00	7 100	....	31 600
	Notched	7 000	4 000	1.71	1.75	10 000	6 000[d]	1.20	1.67	5 000	1.42	

[a] Failed at 75,000,000 to 88,000,000 cycles.
[b] Failed at 246,000,000 cycles.
[c] 1500 psi. below specimen that failed.
[d] Estimated for failure at 100,000,000 cycles.

EQUIPMENT

The machines used for the repeated bending tests were constant-load machines of the Farmer type.[6] The torsion machines[6] used to produce reversals of loads were of the constant-deflection type and were designed in the Fatigue Laboratory of the University of Illinois. The axial load machines[6] were also of the constant-deflection type employing a specially built Moore-Krouse attachment to obtain axial loads in the compressive ranges of the cycles. The speeds of these machines are 1450, 1750, and 1750 rpm., respectively. The tests in corrosion were made by having a small stream of fresh tap water running over the critical section of the specimen.

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[6] For complete description see H. F. Moore and G. N. Krouse, "Repeated Stress (Fatigue) Testing Machines Used in the Testing Laboratory of the University of Illinois," *Circular No. 23*, Engineering Experiment Station University of Illinois, Urbana, Ill. (1934).

The static tension tests were made in a 100,000-lb. Amsler hydraulic testing machine adjusted to a 10,000-lb. or 20,000-lb. capacity. The compression tests were made in a 100,000-lb. Olsen screw-power testing machine, a special compression tool[7] being used to assure axial loading. The static torsion tests were made in a hand-operated pendulum-type machine designed at the University of Illinois and having a capacity of 5000 in-lb.

REPEATED LOAD TESTS

S-N Curves:

A typical set of *S-N* curves is shown in Fig. 4. The stresses plotted for bending and torsion are the nominal maximum stresses at the smallest cross-section and were computed by the formulas $s = \frac{Mc}{I}$ and $s = \frac{Tc}{J}$; no allowance was made for material removed in forming the holes in the notched specimens. For the notched axial load specimens the area, A, at the root of the notch was used to compute the nominal stress from the equation $s = \frac{P}{A}$. For the specimens tested in air, 10 to 20 million cycles of stress were sufficient to determine the endurance limits. In corrosion at least 50 million cycles of stress were used in almost all cases, but (as noted in Table II where all the endurance limits are tabulated) for iron B in particular no stress was reached at which specimens did not fail, even though from 75 to 246 million cycles of stress were applied. In other words, there was no "knee" in any of the *S-N* curves for corrosion-fatigue of iron B, and the *S-N* curves were, in general, straight lines sloping down and to the right. It is realized that in corrosion-fatigue the *S-N* curve may be a horizontal elongated *S*, the increasing downward slope beyond the horizontal portion being due to the fact that corrosion is a function of time and consequently causes more and more damage as time increases. In these tests however, no attempt was made to get a number of cycles larger than that required for the *S-N* curve to become horizontal; that is, to get a "knee," if there was one, in the curve.

The flatness of the curves in Fig. 4 is typical for all the irons tested except iron B. The flatness indicates that any overstressing beyond the endurance limit would lead to an early failure, even though the overstressing were for a short time (small number of cycles) only. An attempt was made to correlate the slopes of the first part of the *S-N* curves with the notch sensitivity of the irons, but except in bending, where the order of steepness was the same as the order for notch sensitivity—steep slope meaning high sensitivity, no correlation could be found. A portion of the difficulty may be ascribed to the small number of specimens sometimes used and the consequent indefiniteness of the slopes of the lines. It should also be noted, see Fig. 4, that the increment of stress between the lowest stressed specimen that failed and the specimen that "ran out," did not fail, was about 1000 psi.; the one exception to this is marked in Table II.

Notch Sensitivity, Mechanical Notches:

Along with the endurance limits, in Table II, are given the notch sensitivity values which are expressed by the stress-concentration factors for repeated loads, K and K_c. K is the ratio of the endurance limit for unnotched specimens tested in air to the endurance limit for notched specimens tested in air; K_c is the ratio of the endurance limit for specimens tested in air to the endurance limit for the same type of specimen tested under a stream of fresh tap water. In other words, K measures the stress-concentration, or damaging effect, due to a mechanical notch (small hole or V-groove) and K_c measures the stress concentration due to a chemical notch (corrosion). The cumulative or total stress-concentration factor due to the combined damaging effect of a mechanical notch plus corrosion is the product of K and K_c.

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[7] W. Leighton Collins, "Compression Testing of Cast Iron," see p. 111.

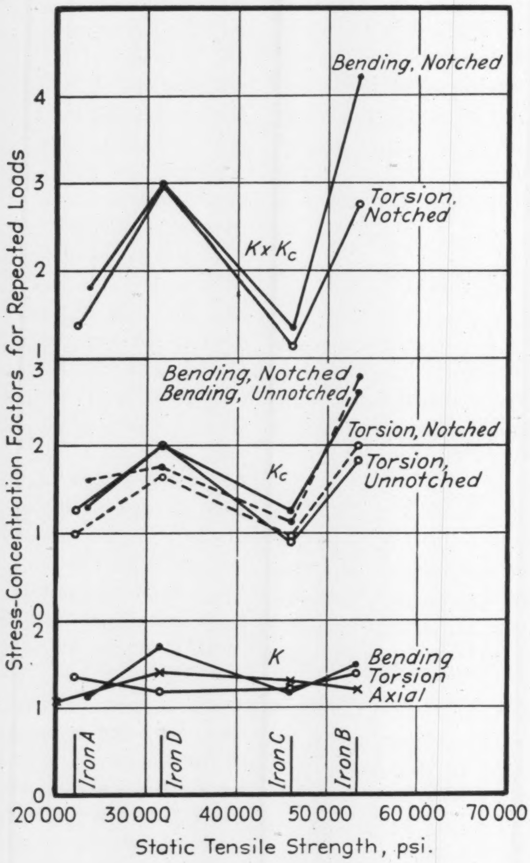

FIG. 5.—Stress-Concentration Factors for Repeated Loads.

$$K = \frac{\text{Endurance limit for unnotched specimens tested in air}}{\text{Endurance limit for notched specimens tested in air}}$$

$$K_c = \frac{\text{Endurance limit for specimens tested in air}}{\text{Endurance limit for similar specimens tested in water}}$$

$K \times K_c$ = Combined effect of stress-concentration factors

To facilitate a comparison of factors

K and K_c and their product for the various irons, the stress-concentration factors have been plotted against static tensile strength in Fig. 5. In considering the stress-concentration factors and the variations in their values, it should be remembered that the increments of stress used to determine the endurance limits usually were 1000 psi. and that such a variation in endurance limit might in some cases cause the stress-concentration factors to vary considerably. The values of K plotted in Fig. 5 indicate that there is a slight trend toward higher values of K as the static tensile strength increases. The average values of K for bending, torsional, and axial loading are as follows: 1.19 for iron A, 1.36 for iron B, 1.23 for iron C, and 1.44 for iron D. The average values of K for all three types of loading and for all four irons is 1.3. These results are, where comparisons can be made, in general agreement with the results of other investigators.[8, 9] Since all failures, regardless of whether the loading was bending, torsional, or axial, were in tension it seems somewhat reasonable that the three respective stress concentration factors for each iron should be about the same, even though the state of stress and distribution of stress are not the same for all three of the above cases of loading.

If, in using the formulas $S = \frac{Mc}{I}$ and $s = \frac{Tc}{J}$ for calculating the bending and torsional stresses, allowance is made for the material removed in forming the hole in computing I and J, the values of K for bending, torsional, and axial loading average 1.09 for iron A, 1.24 for iron B, 1.13 for iron C, and 1.32 for iron D.

Notch Sensitivity, Corrosion Fatigue:

From Fig. 5 it is also seen that the stress-concentration factors for corrosion, K_c, for all irons tested, with the notable exception of iron C, increase with the static tensile strength. The values of K_c for notched and unnotched specimens of any given iron and for any given loading are usually approximately equal; this indicates that the effect of corrosion is independent of the shape of the specimen. The average values of K_c for both bending and torsional loads are as follows: 1.28 for iron A, 2.31 for iron B, 1.07 for iron C, and 1.85 for iron D.

If net cross-sections are used instead of gross cross-sections, as in a previous paragraph, the average values of K_c become 1.12 for iron A, 2.01 for iron B, 0.93 for iron C, and 1.62 for iron D.

The combined effect of the stress-concentration factors K and K_c, which is measured by the product $K \times K_c$, varies in the same general manner as K_c. The values are plotted in Fig. 5 in order that the irons can be compared readily. In bending and in torsion, respectively, the combined effect of stress-concentration factors K and K_c varied from 1.33 and 1.16, for the inoculated iron, C, to 4.20 and 2.76 for the high-strength iron, B. The other irons had intermediate values. Such large variations indicate marked differences in the resistance of the irons to damage caused by corrosion-fatigue combined with the effect of a mechanical notch.

In considering the factor K_c it should be remembered that in some cases, see Table II, no specimens "ran out," that is, did not fail; in such instances the factors K_c are probably too low, but the general comparisons indicated by the remarks made above still apply. In

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[8] J. B. Kommers, "Understressing and Notch Sensitiveness in Fatigue," *Engineering News-Record*, Vol. 109, pp. 353–355 (1932).

[9] A. Pomp and M. Hempel, "Über das Verhalten von Gusseisen und Temperguss unter Wechselnder Beanspruchung," *Mitteilungen*, Kaiser-Wilhelm Institut für Eisenforschung zu Düsseldorf, Band 22, Lief 11, Abhandlung 402, (1940).

order that all factors influencing the results of the tests can be kept in mind, it is also well to recall that the specimens for iron B came from the center of the casting thickness whereas all other specimens came from the quarter point of the thickness and that for iron A the various groups of specimens came from different distances from bottom to top of a casting 57 in. long.

A brief résumé of the data for notch sensitivity, both K and K_c, reveals that the innoculated iron, C, had stress-concentration factors that were almost equal for all types of loads used, that, in general, it was a little less notch sensitive than the more highly alloyed irons B and D, that it resisted corrosion to a much greater extent than either iron B or D and that its values of K and K_c do not vary greatly from those found for ordinary gray iron A.

TABLE III.—ENDURANCE RATIOS.

$$\text{Endurance ratio} = \frac{\text{Endurance limit of unnotched specimens tested in air}}{\text{Static tensile strength of unnotched specimens}}$$

Iron	Static Tensile Strength, psi.	Endurance Ratio			
		Bending	Torsional	Axial	Average
A	22 200[a]	0.38	0.50	0.32	0.35[b]
D	31 600	0.38	0.38	0.22	0.38[b]
C	46 000	0.44	0.37	0.37	0.39
B	53 400	0.39	0.41	0.37	0.39
Avg.	......	0.40	0.39[b]	0.36[b]	....

[a] 23,500 psi. for bending and 20,200 psi. for axial loading because of variation along length of casting.

[b] Extreme values omitted.

Endurance Ratio:

The ratio of the endurance limit for unnotched polished specimens to the static tensile strength of unnotched specimens is defined as the endurance ratio. Values of this ratio for the four irons tested are given in Table III where the irons are arranged in the order of increasing static tensile strength. The endurance ratios for iron A, as noted in Table III, are based on the tensile strength of the iron determined from specimens located at the same sections as the repeated load specimens. The variation in static tensile strength from bottom to top of the long casting of iron A, at intervals of about 5 in., was 25,000, 23,000, 22,100, 22,300, 22,100, 22,100, and 19,300 psi. For the other irons the tensile strengths used are the same as those in Table III. Except for the high ratio, 0.50, for iron A in torsion and the low ratio, 0.22, for iron D in axial loading, most of the values of endurance ratio agree closely in numerical value, although the ratios usually tend to increase slightly as the static tensile strength increases. However, since this variation is so small and since the numerical values for different types of loads varied somewhat, it seems reasonable to state that the average values of endurance ratio for all irons are 0.40 for bending, 0.39 for torsion and 0.36 for axial loads; these averages exclude the two extreme values, one high and one low, previously mentioned.

It is also seen from Table III that the endurance ratios for axial loading are usually a little lower than the ratios for torsion and bending. Since special apparatus and great pains were taken to insure axiality of loading in the compressive range, it is believed that the differences noted are primarily due to the fact that for axial loads the nominal stress over the entire cross-section is constant, whereas in bending and torsion only a small area near the outside of the specimen is stressed to a maximum, or due to the small cross-sectional area of the repeated load specimens for axial loads as compared to the section of the bending and torsional specimens.

Comparison of Endurance Limits:

Because endurance limits for torsional and axial loads are not commonly determined, those endurance limits are expressed in terms of the endurance limits for bending (completely reversed cycles

TABLE IV.—RATIOS OF ENDURANCE LIMITS.
All tests are for unnotched polished specimens tested in air.

Iron	Ratio, Endurance Limit in Torsion to Endurance Limit in Bending	Ratio, Endurance Limit for Axial Load to Endurance Limit in Bending
A	1.22	0.72
B	1.05	0.94
C	0.85	0.85
D	1.00	0.59
Avg.	1.04	0.77

of stress in all cases) in Table IV. Only the data for unnotched polished specimens tested in air are presented because stress-concentration factors, etc., have been discussed separately.

The ratio of endurance limit in torsion to endurance limit in bending varied from 1.22 for iron A to 0.85 for iron C; the average for all four irons is about 1.0. The ratio of endurance limit for axial loads to endurance limit for bending loads varied from 0.94 for iron B to 0.59 for iron D; the average value for all four irons is about 0.8. Since for all three types of loading the failures were in tension it might be expected that both ratios just discussed should have an average value of about unity. The low value of 0.8 for axial loading may be due to the same factors contributing to the low values of endurance ratio for axial loading that have been mentioned previously.

STATIC LOAD TESTS

General:

The static load tests were made in an attempt to study the difference in behaviour of specimens having the same type of notch (circumferential, 60-deg. V-groove 0.062 in. deep and having a radius of 0.01 in. at the root of the notch) under tensile, compressive, and torsional loads and to determine whether values of strength determined in such tests were altered by the presence of the notch.[10] All the static load test specimens had a nominal minimum diameter of 0.500 in. and are shown in Fig. 3.

Data:

The results of all the static load tests, in which only the maximum loads were measured, are given in Table V. Before discussing the data in Table V, however, it is desirable to discuss the types of fracture of the various irons when tested in tension, compression, and torsion.

TABLE V.—STATIC LOAD DATA.
Each value is the average of three tests, except as noted.

$$\text{Stress-Concentration Factor} = \frac{\text{Static Ultimate Strength of Unnotched Specimen}}{\text{Static Ultimate Strength of Notched Specimen}}$$

Iron	Type of Specimen	Stress-Concentration Factor						Ratio, Compressive Strength to Tensile Strength	Ratio, Modulus of Rupture to Tensile Strength
		Tension		Compression		Torsion			
		Strength, psi.	Stress Concentration Factor	Strength, psi.	Stress Concentration Factor	Modulus of Rupture, psi.	Stress Concentration Factor		
A	Unnotched	19 300[a]	....	80 000[a]	....	31 700[a]	....	4.15	1.64
	Notched	21 200	0.91	104 000[a]	0.77	39 100[a]	0.81	4.90	1.84
B	Unnotched	53 400[b]	....	236 000	....	84 100	....	4.42	1.58
	Notched	51 600	1.03	270 000	0.87	88 500	0.95	5.23	1.71
C	Unnotched	46 000	....	140 000	....	62 600	....	3.04	1.36
	Notched	43 800	1.05	193 000	0.73	67 700	0.92	4.40	1.54
D	Unnotched	31 600	....	182 000	....	43 800	....	5.75	1.39
	Notched	32 100	0.98	327 000	0.56	49 600	0.88	10.2	1.54

[a] Average from two tests.
[b] Value from one test; two others were above 47,500 psi.

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[10] J. O. Draffin, Discussion of paper by W. L. Collins and J. O. Smith, "Fatigue and Static Load Tests of a High-Strength Cast Iron at Elevated Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 808 (1941).

Fractures:

In the tension test all fractures were on planes normal to the axis of the specimen, thus indicating a cohesive or separation type of fracture that was due to tensile stress. In the notched specimens the notch, of course, located the section of fracture; the unnotched specimens fractured at random locations in the constant diameter length.

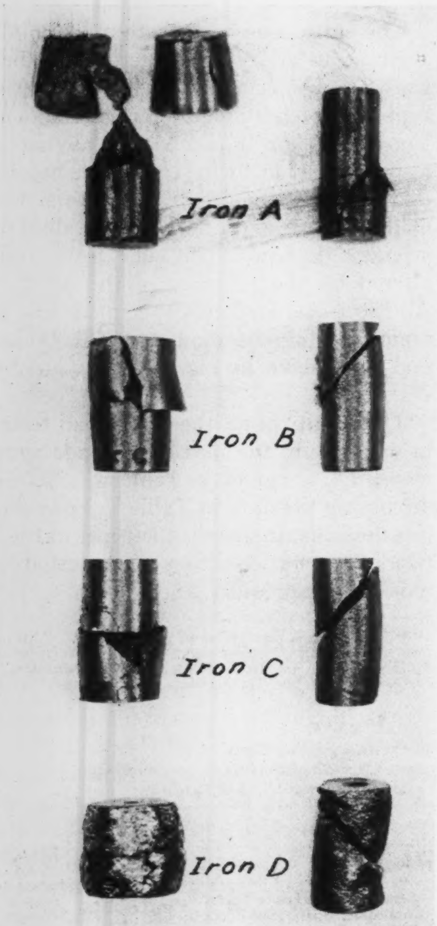

FIG. 6.—Specimens Ruptured by Static Compressive Loads.

The notch was a 60-deg. V-groove 0.05 in. deep and having a root radius of 0.01 in. Nominal minimum diameters of all specimens = 0.500 in.

Compressive loads caused the V-grooves in the notched specimens to close as the load increased, thus increasing (in addition to the "Poisson ratio" effect) the effective cross-sectional area. Consequently the loads resisted by the notched compression specimens were always considerably greater than for the unnotched compression specimens which fractured due to shear on planes making angles of 45 to 60 deg. with the direction of the load. It will be noted, by comparing the ruptured specimens shown in Fig. 6 with the data in Table V, that the greatest ability to undergo large plastic deformation is associated with the highest value of compressive strength of notched specimens and consequently with the lowest stress-concentration factor.

In torsion all the unnotched specimens failed in tension on the usual helicoidal planes. It is interesting to note that the unnotched specimens of iron D twisted through almost 90 deg. in a length of 1.5 in. whereas irons A, B, and C twisted through angles of only about 7, 14, and 19 deg., respectively. These angles of twist correspond to maximum shearing unit strains of about 0.25, 0.05, 0.04, and 0.02 for irons D, A, B, and C, respectively. Static tension tests (not included in this report) of specimens having diameters of 0.800 in. and gage lengths of 8 in. indicated maximum tensile unit strains of 0.0158, 0.0063,[11] 0.0066, and 0.0053 for irons D, A, B, and C, respectively. It is thus apparent that iron D is much more ductile than the other irons and that although the deformation under unidirectional tensile stress is about 1.6 per cent and the fracture is typical of a brittle material, the deformation under a biaxial state of stress, torsion, is about 25 per cent, a magnitude which almost approaches that found for some so-called ductile materials. It is also desirable to note that the type of ductility measured by the static tension and torsion tests does not indicate ability to relieve the high localized stresses that lead to fatigue failure. The notched specimens tested in torsion all failed in shear on the transverse sections through the roots of the notches.

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[11] J. O. Draffin and W. L. Collins, "The Tensile Strength of Cast Iron," *Proceedings*, Am. Soc. Testing Mats., Vol. 37, Part II, p. 88 (1937).

strength. The average for all irons is 0.99, thus indicating that the circumferential V-groove used had practically no effect on the nominal tensile stress developed at fracture. These results are also in close agreement with the conclusions reached by the authors for iron C tested at various elevated temperatures.[12] All of these data and results, however, imply that great care must be taken to insure axial loading.

The stress-concentration factors for static compressive loads have no orderly variation with static ultimate strength in compression. Instead, they seem to vary somewhat as the inverse of the ductility as measured by elongation in the tension test; this already has been discussed under fractures. The closing of the notch before fracture occurred also explains why the factors are all less than unity; the values ranged from 0.56 for iron D to 0.87 for iron B.

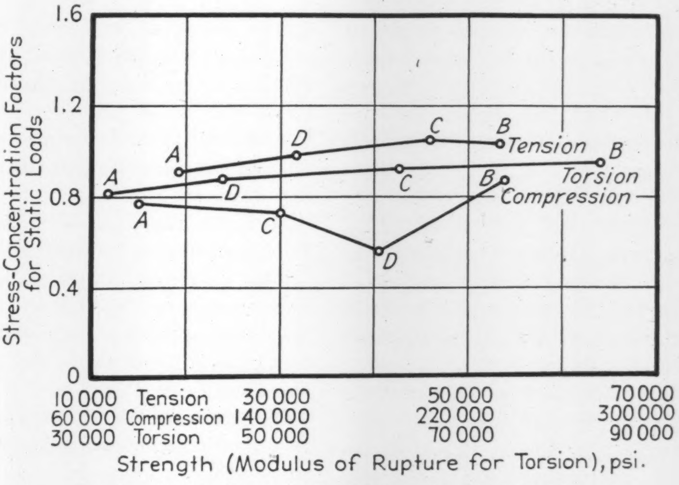

FIG. 7.—Stress-Concentration Factors for Static Loads.

Stress-concentration factor is ratio of strength of unnotched specimen to strength of notched specimen. The letters A, B, C, D refer to the four irons tested.

Notch Sensitivity:

The stress-concentration factors for static loads, that is, the ratio of the static ultimate strength of unnotched specimens to the static ultimate strength of notched specimens of the same iron, are given in Table V and are plotted against static tensile strength in Fig. 7. The variation of this factor for the tension tests is from 0.91 for ordinary gray iron A to 1.05 for the innoculated iron C. In general, the stress-concentration factor tends to increase with static tensile strength.

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[12] W. L. Collins and J. O. Smith, "Fatigue and Static Load Tests of a High-Strength Cast Iron at Elevated Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 797 (1941).

The stress-concentration factors for specimens having a circumferential V-groove and tested in torsion increase as the modulus of rupture for solid, unnotched specimens increases (see Fig. 7). The values increase almost linearly from 0.81 for iron A to 0.95 for iron B. For all irons the values of the stress-concentration factors are less than for tensile loads and greater than for compressive loads. The fact that the stress-concentration factors are all less than unity means that the effect of the notch is not to decrease the load an unnotched specimen of the same minimum diameter can support, but that due to the change in mode of failure from tension to shear the load resisted is increased; the amount of increase, however, decreases as the modulus of rupture of unnotched specimens increases.

The reader should be cautioned that the above values of *K* were obtained for a circumferential 60-deg. V-groove 0.062 in. deep and having a root radius of 0.01 in. If the dimensions of the notch or size of specimen are changed, especially for the compression and torsion specimens, the values of *K* may be changed.

It is worthy of noting that for unnotched specimens the order of increasing strength under static loads is A, D, C, and B for tension and torsion and A, C, D, and B for compression. The only explanation for the difference in order in compression perhaps is that the great ductility of iron D permits more adaptation to accidental eccentricities, or, that the iron just "bulges out" more and thus gives a greater effective or true area of cross-section at failure.

The ultimate strengths in tension, compression, and torsion are compared in the last two columns of Table V. In general, the results of tests by other investigators[13] indicate that as the static tensile strength increases the ratio of compressive strength to tensile strength decreases, but the unordered variation for the four irons tested and reported on herein is probably due to the variety of irons investigated. The ratio of modulus of rupture in torsion, for unnotched specimens, to the tensile strength decreases slightly as the tensile strength increases; the range in values is from 1.64 to 1.36 and this variation is in agreement with the results of tests made by other investigators.[13]

SUMMARY

The purpose of this work was to investigate and compare the notch sensitivity under static and repeated loads of four different cast irons: iron A a slightly alloyed "ordinary" gray iron, iron B a high-strength nickel-molybdenum iron, iron C an innoculated iron, and iron D a high-nickel (austenitic) iron. Due to this variety of materials some of the variations and relationships observed may not apply to *all* cast irons. The conclusions reached are as follows:

Repeated Load Tests:

The repeated load tests consisted of completely reversed cycles of stress due to bending, torsional, and axial loads, respectively. In bending and torsion the notch consisted of a small transverse hole having a diameter 0.10 times the diameter of the specimen; for axial loading the notch was a circumferential 60-deg. V-groove 0.025 in. deep having a radius of 0.01 in. at the root. In bending and torsion both unnotched and notched specimens were also tested under a stream of fresh tap water.

1. The number of cycles of stress required to determine endurance limits for both unnotched and notched specimens

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[13] Cast Metals Handbook, 1940 Edition, p. 366, Am. Foundrymen's Assn.

under a stream of fresh tap water was usually from 30 to 100 million cycles whereas 10 to 20 million cycles were usually sufficient for similar specimens tested in air (see Fig. 4).

2. The average values of the stress-concentration factor K (ratio of the endurance limit for unnotched polished specimens tested in air to the endurance limit for notched specimens tested in air) for repeated bending, torsional, and axial loads, respectively, were as follows: for iron A, 1.19; for iron B, 1.36; for iron C, 1.23; and for iron D, 1.44. The value of K increases with the static tensile strength of the iron, with the exception of iron C which has a relatively low notch sensitivity for an iron of its tensile strength (see Fig. 5 and Table II).

3. The average values of the stress-concentration factor K_c (ratio of the endurance limit for specimens tested in air, either notched or unnotched to the endurance limit for similar specimens tested under a stream of fresh tap water) for repeated bending, torsional, and axial loads were as follows: for iron A, 1.28; for iron B, 2.31; for iron C, 1.07; and for iron D, 1.85. The value of K_c increases with the static tensile strength of the iron with the exception of iron C which has a relatively low notch sensitivity for an iron of its tensile strength.

4. The stress-concentration factors for corrosion-fatigue of notched specimens, K_c, were usually within ±10 per cent of the values of K_c found for unnotched polished specimens (see Fig. 5 and Table II).

6. The ratio of endurance limits for unnotched polished specimens tested in air to static tensile strengths for all irons and for all the loads used can be considered to be 0.4. In only three cases were differences from 0.4 greater than ±10 per cent (see Table III).

7. The ratio of endurance limit for unnotched polished specimens in torsion to the endurance limit for similar types of specimens in bending, all tests being in air, varied from 1.22 for iron A to 0.85 for iron C; the average value for all irons is about 1.0 (see Table IV).

8. The ratio of endurance limit for unnotched polished specimens subjected to axial loads to endurance limit for similar types of specimens in bending, all tests being in air, varied from 0.59 for iron D to 0.94 for iron B, the average value for all irons being about 0.8 (see Table IV).

Static Load Tests:

All of the specimens used had a nominal minimum diameter of 0.500 in. In the notched specimens the notch was a circumferential 60-deg. V-groove, 0.062 in. deep and having a radius of 0.01 in. at the root.

9. The notch used caused the fracture in the torsion test to change from tension to shear.

10. The presence of the notch had practically no effect on the static tensile strength, although the stress-concentration factor (ratio of the tensile strength of unnotched specimens to the tensile strength of notched specimens) did increase slightly as the tensile strength of the iron increased; the average value is unity (see Fig. 7 and Table V).

11. There was no relation between the stress-concentration factor in compression (ratio of the compressive strength of unnotched specimens to the compressive strength of notched specimens) and the ultimate compressive strength. The unit load supported by the notched specimens was greatly influenced by the ductility of the material permitting the notch to close and thus increasing the effective cross-sectional area (see Fig. 7 and Table V).

12. The stress-concentration factor in torsion (ratio of the modulus of rupture

of unnotched specimens to the modulus of rupture of notched specimens) increased from 0.81 to 0.95 as the modulus of rupture increased (see Fig. 7 and Table V).

13. There was no consistent relationship between ultimate strengths in compression and tension for unnotched specimens; the values of their ratios varied from 3.04 for iron C to 5.75 for iron D (see Table V).

14. The modulus of rupture in torsion of unnotched specimens was very nearly 1.5 times the static tensile strength of unnotched specimens. The values varied from 1.36 for iron C to 1.64 for iron A (see Table V).

Acknowledgment:

All of the testing was performed in the laboratories of the Department of Theoretical and Applied Mechanics as a project of the Engineering Experiment Station of the University of Illinois. The authors acknowledge the cooperation of the three companies that furnished irons B, C, and D, the help of R. P. Molt, W. J. Shive, S. J. Capizzi, and L. E. Barnes, seniors in the College of Engineering and C. M. Middlesworth, Graduate Student, who aided with the testing, and of J. T. MacKenzie of the American Cast Iron Pipe Co. for furnishing the photomicrographs and the chemical analyses.

DISCUSSION

MR. T. MCLEAN JASPER[1] (*by letter*).—The writer is keenly interested in the principle of testing applied by the authors in arriving at the notch sensitivity of various materials. It is believed that the fatigue notch result comparisons are directly applicable to dynamic loading of structures and that the static notch result comparisons are directly applicable to static structures. For many years practical engineers have become increasingly aware that the notch impact bending results could be applied only with a great degree of caution and that it was a test of questionable value which could "not be used for the quantitative comparison of different types of materials. . . .In this sense the test is to be regarded as little more than qualitative so that only the larger differences between results are important."[2]

With experience in the fatigue and static notch comparative type of testing, it is believed that engineers will obtain much help in the application of materials to structures, and although this method of testing is not new to us, the authors have given us valuable information on cast iron of various strengths. It is to be hoped that more tests of this type will be available. The question of relative size of specimen and notch may be important.

With reference to the test results, we have, in general, felt that a notch was more detrimental to cast iron than the results show. Again, tests are more valuable than deductions from tests on steel. In the curve marked K_c is added the effect of corrosion-fatigue which is an appropriate way of applying this factor. I am wondering about the curves obtained by multiplying K_c by K. How do the authors think this applies?

I wish to compliment the authors on making available valuable information to our engineering staffs and hope that by so doing they may become expert advisers to our production groups.

MR. H. F. MOORE[3].—I should like to offer a comment in connection with the tension test results of the cast iron. Mr. Collins found that he had a stress-concentration factor of about unity. That, to my mind, is an indication, first, that he was very careful in centering his tension specimens, and, second, that the form of tension specimen, as shown on page 644, which is a form often used at the University of Illinois, is a pretty good form of specimen. I do not know of a much more difficult problem in testing than to get a tension specimen, especially of a nonductile material, in which the actual stress distribution does not show a maximum stress of at least 10 per cent above the mean. There are data in English tests, and other tests in this country, to support that statement. I believe this specimen with its very gradual reduction of diameter, cut with a tool swung on radius, is quite satisfactory. If I were asked to determine the tensile strength of cast iron and were allowed to choose my own method, I should make a torsion test on a hollow circular specimen. Cast iron is funny stuff; I do not know how you get the shearing strength of it unless in a compression test. The tensile strength you get directly, and can get it from a torsion test of a hollow specimen, and possibly from a test of an I-beam shaped flexural specimen with thin edge and flanges.

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[1] Director of Research, A. O. Smith Corp., Milwaukee, Wis.

[2] This is quoted from Mr. Rosenhain's summary as chairman of Group A and applied to the Notch Bar Impact Test Symposium of the International Association for Testing Materials at Zurich, 1931.

[3] Professor of Engineering Materials, University of Illinois, Urbana, Ill.

MR. W. LEIGHTON COLLINS[4] (*author's closure*).—The authors greatly appreciate the kind comments of Mr. Jasper relative to their work and appreciate the suggestions made for the practical application of the data herein reported. The question about the use of the curves $K \times K_c$ of Fig. 5 can be answered best by saying that $K \times K_c$ is a measure of the stress concentration factor (damage) caused by the particularly damaging effect of a mechanical notch and corrosion. Such conditions are not unknown in industrial equipment and, in such cases, the product of K and K_c would be used as other stress concentration factors in formulas such as $s = (K \times K_c)\frac{T_c}{J}$. To use the numerical values in the paper would, of course, mean that the corrosive medium, type of notch, etc., would have to be the same as those used in the tests.

Mr. Moore's statements regarding methods of determining various static load properties of cast iron emphasize the need for more testing if the properties of cast iron are to be understood fully. The author has made quite a number of torsion tests of hollow cast iron specimens, and, as Mr. Moore suggests, they did fail in tension.[5] However, the problem is not as simple as it may seem because the modulus of rupture in torsion is a function of the ratio of wall thickness to outside radius for a wide range of values.

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[4] Assistant Professor, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[5] Jasper O. Draffin and W. L. Collins, "Effect of Size and Type of Specimen on the Torsional Properties of Cast Iron," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part II, p. 235 (1938).
Jasper O. Draffin and W. L. Collins, "Mechanical Properties of a High-Strength Cast Iron," *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 589 (1939).

SOME CREEP STUDIES ON CUPOLA MALLEABLE CAST IRON

By J. J. Kanter[1] and Glen Guarnieri[1]

Synopsis

In view of the apparent lack of creep information on malleable irons, a series of high-temperature studies on cupola malleable cast iron are reported. These studies seem to indicate that this material makes a very creditable showing in relation to the accepted values for silicon-killed carbon steel, having two-thirds creep strength at 800 F. The allowable working stress for malleable iron, based upon creep studies using an established policy of determining design stress, justifies 5600 psi. up to 775 F., a value several times that used in current designs limited to 450 F. maximum service temperature. Long-time fracture tests at high temperature, as well as tension tests at room temperatures on specimens with creep test history, and micrographical studies indicate excellent stability for malleable iron. Studies of permanent growth incident to creep testing show little effect until temperatures of 900 F. are reached, and then only a slight growth attributable in part to graphitization of residual pearlite. The studies indicate, on the whole, that current temperature limitations placed upon the use of malleable iron may be too conservative.

It appears that few, if any, data upon the elevated temperature creep properties of malleable cast irons have been made available. Although a few references[2] may be cited to short-time tensile strength values at temperatures up to 1200 F., no tensile creep strength values for malleable iron seem to have gained acceptance.[3] This lack of information upon which allowable working stress values for malleable irons might be based at various temperatures may have hindered engineers in taking full advantage of this material in high-temperature applications. Indeed, the rather unjustified reluctance on the part of engineers to consider malleable iron as a high-temperature material might explain its oversight among the extensive creep researches which have extended over all manners of metals and alloys.

When rating malleable iron for temperature service, particularly for pressure-containing parts such as pipe fittings, engineers seem to have categorically placed malleable irons in the same bracket as gray iron and entirely dismissed it from consideration in favor of steels and expensive alloys. No manufacturer of malleable iron flanges, pipe fittings, or valve parts has stated any ratings for temperatures higher than 550 F. and safety and inspection codes are even more restrictive at 450 F.

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[1] Materials Research Engineer, and Metallurgical Engineer, respectively, Crane Co., Chicago, Ill.

[2] Symposium on Effect of Temperature on the Properties of Metals, published jointly by the American Society for Testing Materials and The American Society of Mechanical Engineers (1931). (Issued as separate publication.)

[3] A paper read before the Malleable Iron Division of the American Foundrymen's Assn., Chicago, Ill., June 22, 1933, by H. W. Maack, "High Temperature Tests of Malleable Iron," made reference to a preliminary survey of creep strength of malleable iron,—some of the data are incorporated in Table III of this paper.

PRINT IN BIND

Perhaps in these times when the available steel tonnage and capacity for its production and fabrication are short of fulfilling urgent requirements, a critical examination of the properties of malleable iron at high temperatures, with a view to widening its use as a substitute for steel (as well as brass, bronze, and nickel-copper alloys) at temperatures above the 450 to 550 F. limits, might prove fruitful.

MATERIAL AND TEST METHODS

A series of creep studies upon cupola malleable cast iron are herein presented, along with certain other data pertinent to a critical evaluation upon metallurgical principles of the high-temperature usefulness of this product. The material used in these studies is identical with that reported upon by Ziegler, Meinhart and Deacon in their discussion of graphitization.[4]

TABLE I.—SHORT-TIME TENSILE PROPERTIES OF CUPOLA MALLEABLE IRON.

Specimen[a]	Temperature, deg. Fahr.	Tensile Strength, psi.	Proportional Limit, psi.	Yield Stress (0.2 per cent set), psi.	Modulus of Elasticity, psi.	Elongation in 2 in., per cent	Reduction of Area, per cent
457–49	80	41 500	21 150	30 500	23.8×10^6	9	8
457–45	500	39 000	18 000	26 950	24.5×10^6	6	5
457–50	600	38 700	17 950	25 500	20.8×10^6	5.5	5
457–48	800	33 200	14 425	23 000	22.5×10^6	5.5	5
457–62	1000	16 150	9 300	14 500	16.0×10^6	10.0	11

[a] All specimens machined to 0.505 in. in diameter.

The composition of the white iron from which these bars were made was reported as follows:

Silicon, per cent	0.91
Manganese, per cent	0.47
Sulfur, per cent	0.092
Phosphorus, per cent	0.17
Graphitic carbon, per cent	0.18
Combined carbon, per cent	2.98
Total carbon, per cent	3.16

After a malleablizing treatment according to regular manufacturing practice along with many other castings, the total carbon was found to be 2.78 per cent, of which 0.005 per cent is estimated from microstructure to be combined as pearlite.

Tension test bars were cast and treated in accordance with A.S.T.M. Standard Specifications for Cupola Malleable Iron (A 197 – 39),[5] which requires a 7½ in. long specimen with gripping ends ¾ in. in diameter and a breaking section ⅝ in. in diameter by 2½ in. long. These were machined to specimens with 0.505-in. diameter breaking sections with ¾-in. standard threaded ends suitable for both short and long-time tension tests. Tension tests were made in accordance with the A.S.T.M. Tentative Method of Test

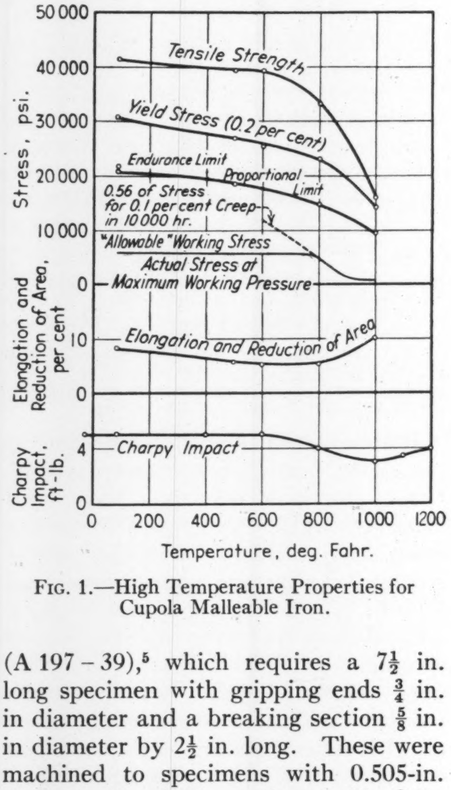

FIG. 1.—High Temperature Properties for Cupola Malleable Iron.

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[4] N. A. Ziegler, W. L. Meinhart and A. J. Deacon "Graphitization of Cementite in Cupola White Iron," *Transactions*, Am. Foundrymen's Assn., Vol. 49, No. 3, March, 1942, pp. 449–479.

[5] 1939 Book of A.S.T.M. Standards, Part I, p. 502.

BINDING

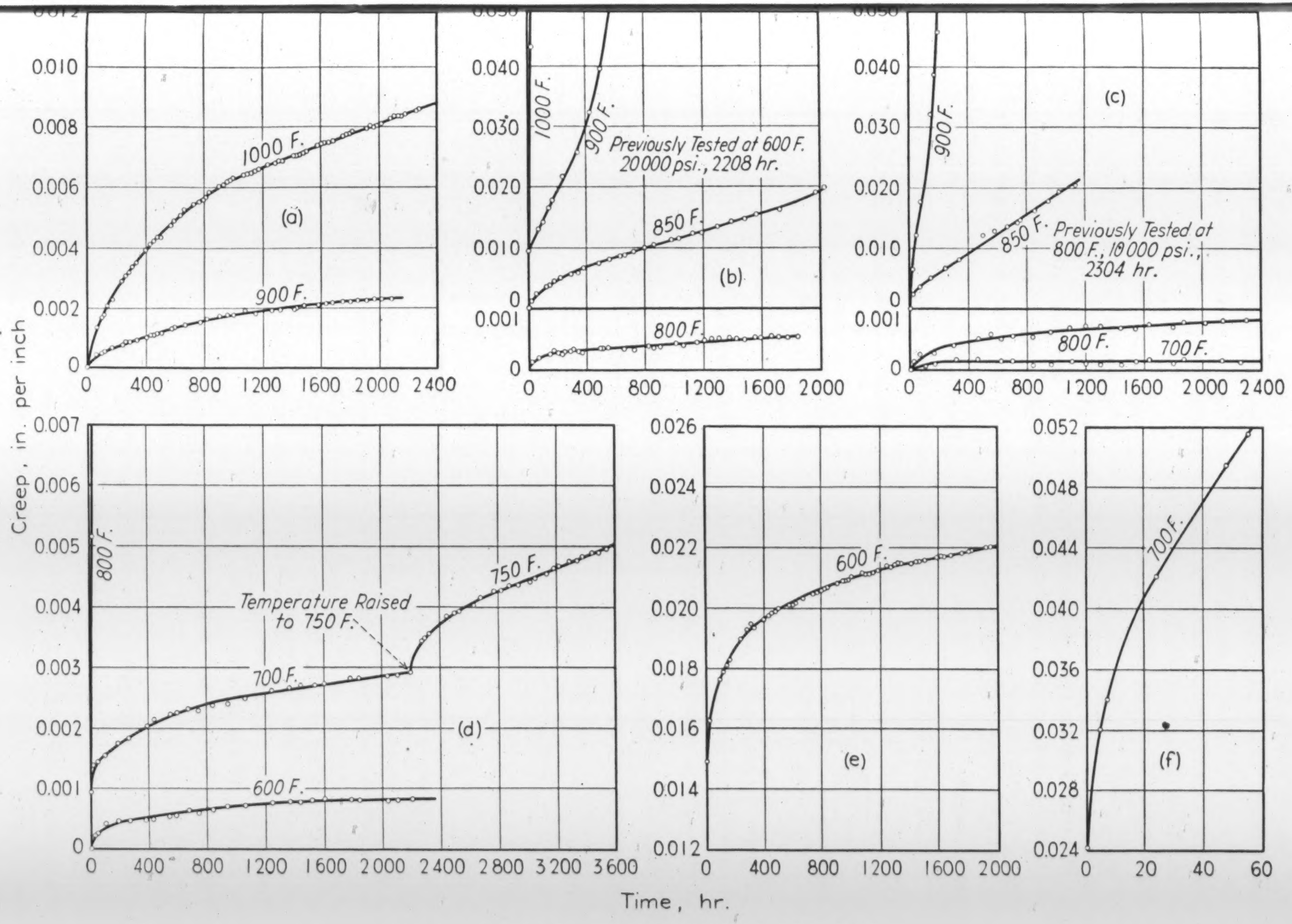

FIG. 2.—Deformation *versus* Time for Cupola Malleable Iron.

(*a*) At 3000 psi. load. (*b*) At 7500 psi. load. (*c*) At 10,000 psi. load. (*d*) At 20,000 psi. load. (*e*) and (*f*) At 30,000 psi. load.

for Short-time High-Temperature Tension Tests of Metallic Materials (E 21 – 37 T)[6] and Standard Recommended Practice for Conducting Long-Time High-Temperature Tension Tests of Metallic Materials (E 22 – 41).[7]

SHORT-TIME TENSILE PROPERTIES

The tensile properties of the iron used in these investigations as determined from the unmachined bars as called for in the A.S.T.M. Specifications A 197 are as follows:

	Tensile Strength, psi.	Yield Point, psi.	Elongation in 2 in., per cent
Test values	43 500 to 49 000	33 800 to 35 000	6.0 to 7.0
Specification requirements	40 000 min.	30 000 min.	5 min.

In order satisfactorily to determine the elevated temperature short-time tensile properties, using an extensometer, it was necessary to use machined 0.505-in. diameter bars. These results are given in Table I, where it is to be noted that removal of the cast skin has somewhat lowered the strength. These results are plotted in Fig. 1, including values for Charpy impact strength as measured by the keyhole notched bar. Due to the approximate numerical equivalence of the elongation and reduction of area percentages, these values are both represented by a single curve.

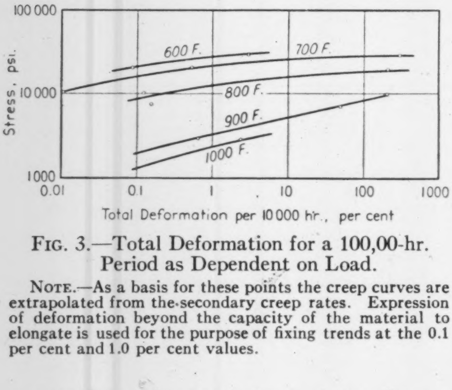

FIG. 3.—Total Deformation for a 100,00-hr. Period as Dependent on Load.

NOTE.—As a basis for these points the creep curves are extrapolated from the secondary creep rates. Expression of deformation beyond the capacity of the material to elongate is used for the purpose of fixing trends at the 0.1 per cent and 1.0 per cent values.

CREEP DATA

The results of 15 creep tests are here reported. The individual creep-time curves for these tests are shown in Fig. 2. These curves, representing a total of about 23,000 hr. of testing, are the basis of the total deformation estimates per 10,000 hr. of Fig. 3. The stress-deformation curves of Fig. 3 are slightly on the conservative side of curves derived for rate of creep; due allowance is thereby made for the slight permanent growth incident to the creep of this material as shown further on. Stresses estimated to produce 0.1 per cent and 1.0 per cent total creep in 10,000 hr. at various temperatures read from the curves of Fig. 3 are given in Fig. 4.

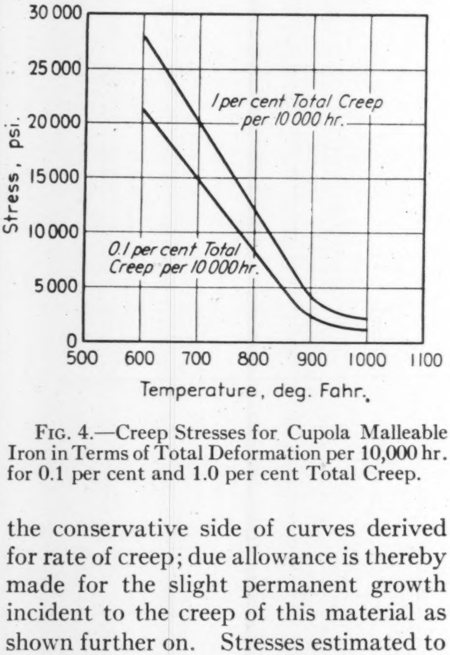

FIG. 4.—Creep Stresses for Cupola Malleable Iron in Terms of Total Deformation per 10,000 hr. for 0.1 per cent and 1.0 per cent Total Creep.

ALLOWABLE WORKING STRESS

It is of interest to compare these creep stresses for malleable cast iron with the allowable working stresses established by the A.S.M.E. Boiler Construction Code[8] for silicon-killed carbon steels having 70,000 psi. minimum tensile strength. The stresses there given for ferrous materials at high temperatures are in general based upon 80 per cent of the accepted creep strength (based on 1 per cent deformation per 100,000 hr.) for each grade of material recognized. Where castings are involved, the Code applies a further 70 per cent quality factor. For the sake of a comparison, let us consider 80 per cent of the 0.1 per cent per 10,000 hr. total creep values for cupola malleable iron for the range 800 to 1000 F., to which is further applied a 70 per cent quality factor since we are dealing with castings. The "Allowable" working stress curve of Fig. 1 is illustrative of this procedure. In deriving allowable stresses for the Boiler Code purposes a policy is followed, in the case of carbon steel castings, of permitting 70 per cent of one fifth of the minimum specified tensile strength to be used at all temperatures from −20 F. up to 650 F. Above 650 F. this tensile stress curve is faired with the discounted creep stress curve. Suppose this same policy were to be followed in picking allowable design stresses for 40,000 psi. minimum tensile strength cupola malleable iron, then the allowable stress would be 5600 psi., a value which fairs with the discounted creep stresses in Table II for cupola malleable iron at 775 F. At 775 F., the creep strength of the cupola malleable iron is found to be about 67 per cent as great as that accepted for silicon-killed carbon steel. Bursting tests on malleable iron pipe fittings indicate that existing designs are actually rated to operate under temperature at stresses well within 5600 psi. (For example: a 2 in. tee rated for 300 psi. working pressure, bursts at 7000 psi. hydraulic pressure, manufactured from 44,000 psi. tensile strength malleable iron. Therefore, working stress is approximately $44{,}000 \times \frac{300}{7000}$ or 1886 psi., a 3:1 factor upon 5600 psi.).

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[6] 1939 Book of A.S.T.M. Standards, Part I, p. 1254.

[7] 1941 Supplement to Book of A.S.T.M. Standards Part I, p. 206.

TABLE II.—ALLOWABLE WORKING STRESS.

Temperature, deg. Fahr.		Stress, psi.		Ratio
		Cupola Malleable Iron	Carbon Cast Steel	
600	Based upon tensile strength	5600	9800	0.57
650		5600	9800	0.57
700		5600	9300	0.60
750		5600	8330	0.67
800	Based upon creep data	4760	7000	0.68
850		2850	5460	0.52
900		1340	3920	0.34
950		1050	2660	0.41
1000		722	1440	0.50

TABLE III.—DUCTILITY OF FRACTURED CREEP SPECIMENS OF CUPOLA MALLEABLE IRON.

Specimen	Temperature, deg. Fahr.	Stress, psi.	Time for Fracture, hr.	Elongation, per cent	Reduction of Area, per cent
457–42	700	30 000	62	7.0	6.0
457–36	800	20 000	207	7.0	7.5
190–12[a]	800	20 000	144	8.0	17.1
457–35	900	7 500	840	7.5	9.5
457–34	900	10 000	456	23.0	16.0
190–10[a]	900	10 000	844	10.5	12.2
457–41	1000	7 500	29	10.5	11.0
190–11[a]	1200	3 000	264	15.0	7.0

[a] These specimens were preloaded at lower temperatures as follows:
190–12, 550 F. for 192 hr., 600 F. for 120 hr., 650 F. for 816 hr., 700 F. for 864 hr., Total 1992 hr.
190–10, 550 F. for 144 hr., 600 F. for 120 hr., 650 F. for 120 hr., 700 F. for 96 hr., 750 F. for 312 hr., 800 F. for 120 hr., 850 F. for 984 hr., Total 1892 hr.
190–11, 800 F. for 144 hr., 850 F. for 168 hr., 900 F. for 192 hr., 950 F. for 432 hr., 1000 F. for 1008 hr., 1050 F. for 1200 hr., 1100 F. for 480 hr., 1150 F. for 1344 hr., Total 4968 hr.

STABILITY OF PROPERTIES

Lest it be argued that cupola malleable iron is not stable in its properties under creep conditions, Table III is given to demonstrate its high temperature ductility-to-fracture. Not only is the normal capacity to elongate retained, but at

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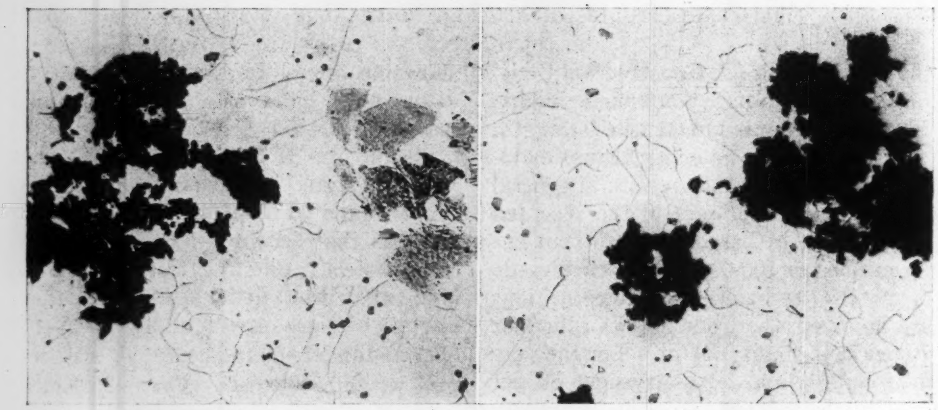
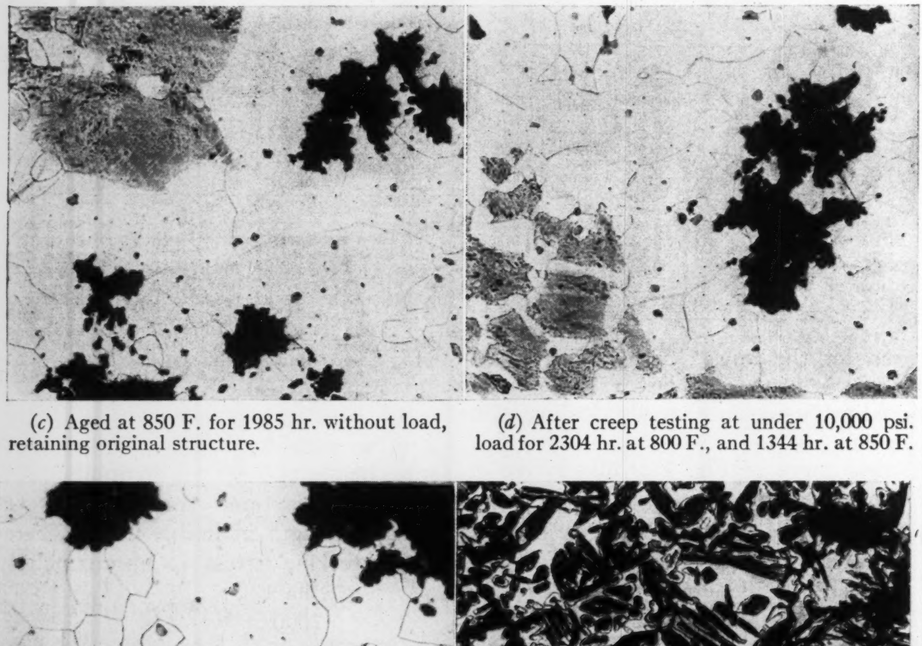
[8] Addenda to A.S.M.E. Boiler Construction Code September 30, 1941, Tables U2 or P7.



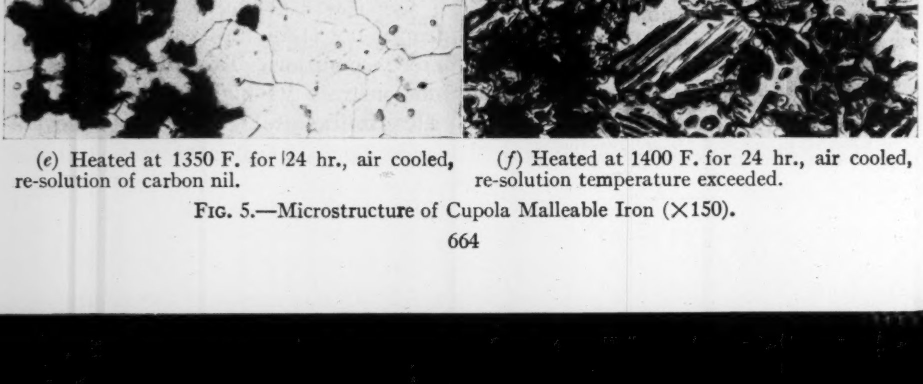
(*a*) Original structure showing graphite nodules and residual pearlite.

(*b*) After creep testing at 1000 F. under 3000 psi. load for 2280 hr., residual pearlite graphitized.



(*c*) Aged at 850 F. for 1985 hr. without load, retaining original structure.

(*d*) After creep testing at under 10,000 psi. load for 2304 hr. at 800 F., and 1344 hr. at 850 F.



(*e*) Heated at 1350 F. for 24 hr., air cooled, re-solution of carbon nil.

(*f*) Heated at 1400 F. for 24 hr., air cooled, re-solution temperature exceeded.

FIG. 5.—Microstructure of Cupola Malleable Iron (×150).

the higher temperatures a considerable increase in ductility is found, indicating excellent time-to-fracture strength.

The capacity of cupola malleable iron to elongate under the influence of stress, time, and temperatures, even at 1200 F. after an exposure of more than 5000 hr., gives pause to reflect upon the stability against intergranular oxidation conferred upon iron and steel by silicon content. It has been well established in time-to-fracture tests of steels that silicon is a specific to correct tendency toward "short" high-temperature breaks, due to intergranular oxidation. It may well be that it is to the silicon content of malleable iron that its capacity for sustained high-temperature ductility is attributable.

TABLE IV.—ROOM TEMPERATURE TENSILE PROPERTIES OF CREEP TEST SPECIMENS OF CUPOLA MALLEABLE IRON.

Specimen	Creep Test Temperature, deg. Fahr.	Creep Test Stress, psi.	Creep Test Time, hr.	Room Temperature Properties				
				Tensile Strength, psi.	Proportional Limit, psi.	Yield Stress (0.2 per cent set), psi.	Total Elongation, per cent[a]	Reduction of Area, per cent
457-40	600	30 000	1944	38 900	35 700	36 650	6.5	6
457-32	700	20 000	2208	39 000	24 950	31 400	6.5	5
	750	20 000	1416					
457-43	800	7 500	1834	41 000	17 500	29 750	10.0	8
457-46	900	3 000	2088	36 800	18 900	23 900	7.0	5
457-33[b]	1000	3 000	2280	Charpy Impact = 4.5 ft-lb.				

[a] Includes elongation from creep test.

[b] Undersized Charpy specimen machined from gage length of creep specimen.

Several tension tests, as made upon specimens with creep test history, are shown in Table IV. It is evident from these results that at atmospheric temperature after stress and temperature exposure malleable iron continues to exhibit good physical properties. The microstructures for some of the test specimens used are shown in Fig. 5. Again there seems to be little change from the original structure, except for the evidences of working in the overstressed specimens. At the highest testing temperature represented in these micrographs, 1000 F., no evidence of re-solution of the carbon is found. On the contrary, complete graphitization of the residual pearlite has occurred. In Table V is shown a series of heating tests made to indicate the temperature of re-solution of the carbon in the iron used in these creep studies. Apparently no appreciable solution occurs until a temperature of 1350 F. is exceeded, as indicated by the hardness measurements and micrographs (Fig. 5(*e*) and (*f*)). Thus, re-solution of carbon in the range of temperature for the creep studies does not seem to be a factor of consequence.

TABLE V.—HEAT TREATMENTS TO DETECT GRAPHITE SOLUTION OF CUPOLA MALLEABLE IRON.

Specimen	Hardness Before Treatment Vickers Pyramid Number at 30-kg. Load	Temperature, deg. Fahr.	Time at Temperature, hr.	How Cooled	Hardness After Treatment, Vickers Pyramid Number at 30-kg. Load
457-37-A	126	1175	24	Air	124
457-37-W	125	1175	24	Water quenched	143
457-40-A	124	1200	24	Air	124
457-40-W	122	1200	24	Water quenched	132
457-47-A	124	1225	24	Air	121
457-47-W	120	1225	24	Water quenched	135
457-48-A	120	1250	24	Air	122
457-48-W	121	1250	24	Water quenched	138
457-49-A	122	1275	24	Air	128
457-49-W	122	1275	24	Water quenched	126
457-42-A	114	1300	24	Air	118
457-42-W	116	1300	24	Water quenched	134
457-50-A	119	1325	24	Air	128
457-50-W	116	1325	24	Water quenched	141
457-46-A	113	1350	24	Air	121
457-46-W	117	1350	24	Water quenched	146
457-38-A	116	1400	24	Air	163
457-38-W	115	1400	24	Water quenched	310

Neither do the dynamic properties of malleable iron appear to be impaired by temperature as judged by Charpy keyhole notched-bar impact tests. From

−25 F. up to well above 600 F. the same values as for room temperature are found, a slight decrease commencing at about 800 F. The endurance limit of this material as shown in Fig. 6 was found to be in the neighborhood of 22,000 psi. The fatigue tests were made on an R. R. Moore high-speed machine. These results are here given in this connection, because a review of the literature failed to reveal any endurance limit values for cupola malleable iron. In view of the magnitude of allowable working stress which would be permitted for malleable iron castings using a policy similar to that established by the A.S.M.E. Boiler Construction Code, an endurance limit of 22,000 psi. leaves a comfortable margin of safety,—the ratio between 22,000 and the 5600 psi., derived above, being approximately 4:1.

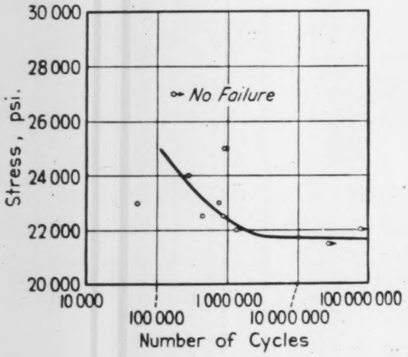


FIG. 6.—*S-N* Diagram for Cupola Malleable Iron at 80 F. Indicating an Endurance Limit of About 22,000 psi. Stress.

PERMANENT GROWTH

An attempt has been made to estimate the permanent growth of cupola malleable iron incident to the creep deformation. Where growth has occurred, therefore, the curves of Fig. 2 do not represent true creep, but instead a summation of extension due to both growth and creep. Measurements have been made upon certain of the specimens after removal from the test furnaces to differentiate between the actual creep deformation and the amount of growth which occurred.

A creep specimen which has undergone only plastic extension, but no change in volume, would (from purely geometrical considerations) be expected to have undergone a unit lateral contraction exactly one half as great as the unit extension. Should the unit lateral contraction be *less* than one half of the unit extension, it is to be concluded that the specimen has sustained an increase in volume. Assuming that the volume increase or growth is isotropic, it can be shown that linear growth may be estimated from the total length change and the diametral contraction by the formula:

$$L_G = \frac{2D_T + L_T}{3}$$

where

L_G = length change in creep specimen due to growth in per cent,

D_T = difference between final and original diameter in per cent, and (contraction expressed with negative sign), and

L_T = total length change in specimen due to creep and growth in per cent.

In Table VI are given the linear growth estimates for cupola malleable iron which could be made by this method from the creep specimens of the present investigation. Several growth values for the unstressed condition at various temperatures are given for comparison, some of which were previously reported by one of the authors in the 1931 Symposium on Effect of Temperature on the Properties of Metals.[9]

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[9] Discussion by J. J. Kanter, p. 459.

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Where structural changes in the iron ccur, as in the case of growth through raphitization, the creep rate itself is ccelerated and conversely there is defiite evidence to indicate that strains romote processes of diffusion and thus ccelerate growth. The data of Table I indicate that growth in the absence of train is not a factor at temperatures nder 900 F. However, under the excesive straining imposed by loads in excess of the yield strength, growth is observed t 600 F. It appears, though, that no onsequential growth occurs up to 800 F. t stresses of the magnitude of interest or design purposes.

ABLE VI.—PERMANENT GROWTH OF CUPOLA MALLEABLE IRON.

Specimen	Temperature, deg. Fahr.	Stress, psi.	Time of Test, hr.	Growth, per cent	Total Deformation, per cent
931 Symposium[a]	600	0	20 500	0	....
457–35	600	20 000	2 208	0.03	0.084
457–40	600	30 000	1 944	0.20	2.20
457–32	700	20 000	2 208	0	0.29
931 Symposium[a]	750	0	11 600	0.02	....
457–38	750	10 000			
457–32	750	20 000	1 416	0.13	0.21+ (previous test 0.29)
457–43	800	7 500	1 834	0.01	0.052
457	850	0	1 985	0.07	....
457–44	850	7 500	2 376	0.46(±0.10)	4.16
457–39	800 850	10 000 10 000	2 304 1 294	0.31	2.20
931 Symposium[a]	900	0	20 500	0.26	....
457–46	900	3 000	2 088	0.07	0.228
457–33	1000	3 000	2 280	0.40	0.359

[a] Symposium on Effect of Temperature on the Properies of Metals, published jointly by the American Society or Testing Materials and The American Society of Mehanical Engineers (1931). (Issued as a separate ublication.)

SUMMARY AND CONCLUSIONS

1. These studies indicate that at 800 F. upola malleable iron has about two hirds of the creep strength of siliconilled carbon steel at that temperature. bove 800 F. and up to 1000 F. the ratio etween malleable iron and steel varies etween one half and one third.

2. Were an allowable working stress for cupola malleable iron to be based upon these data, using a similar policy to that followed by A.S.M.E. Boiler Code, a stress of 5600 psi. is indicated up to 775 F. This stress value is two to four times as great as used in present designs of pipe fittings, limited by codes to 450 F. maximum service temperature.

3. Long-time fracture tests at high temperature indicate that the ductility of cupola malleable iron is unimpaired. Actually greater ductility to fracture is observed in the 900 to 1200 F. range than for tests at room temperature, indicating excellent time-to-fracture strength.

4. Tension tests upon specimens with creep test history at temperatures up to 1000 F. show that good physical properties are retained. The only significant structural change noted micrographically is a tendency to complete graphitization of residual pearlite.

5. Data from short-time tension tests, Charpy keyhole notched-bar impact tests, and endurance limit determinations for cupola malleable iron of the composition used for creep studies give a background for interpretation.

6. No growth occurs up to 800 F. at stresses of the magnitude of interest for design purposes. At 900 F. and above, a slight growth appears, attributable in part to graphitization of residual pearlite.

7. The high temperature studies of cupola malleable iron indicate that the current engineering temperature limits upon the use of this material may be too conservative.

Acknowledgments:

The authors wish to acknowledge help and advice rendered in the preparation of this paper by A. M. Houser, Sr., J. P. Magos, and associates in the Crane Research Laboratories,—also to A.S.T.M. Committee A-7 on Malleable-Iron Castings for its resolution that the preparation of a paper on the creep tests of malleable iron be solicited.

THE CREEP CHARACTERISTICS OF SOME COPPER ALLOYS AT ELEVATED TEMPERATURES

By H. L. Burghoff,[1] A. I. Blank,[1] and S. E. Maddigan[1]

Synopsis

This paper presents the results of creep tests upon seven wrought copper alloys at 300, 400, and 500 F. The test materials included 70-30 brass, silicon bronze (3 per cent silicon), 85-15 brass, naval brass, admiralty brass, phosphor bronze (5 per cent tin), and 70-30 copper-nickel. These were prepared in various tempers to permit evaluation of the effect of grain size in annealed tempers and of degree of reduction in drawn tempers. The 70-30 brass was investigated in more detail than were the other alloys.

The tests were made on samples of 0.125 in. diameter wire, with gage length of 10 in., in three forced convection, multiple-specimen, furnaces. Duration of most of the tests was of the order of 5000 hr., although some tests of special interest were run well beyond this time. The character of some of the creep curves which were obtained demonstrates the wisdom of extending creep testing periods as long as possible.

Grain size and degree of cold working are important factors in determining the creep resistance of these alloys, and in general are fully as important as chemical composition. The creep resistance of the hard-worked alloys is largely determined by their recrystallization characteristics. If recrystallization takes place over a prolonged period, the materials may be very unstable with regard to creep.

The 70-30 copper-nickel alloy is easily the best of the alloys tested for service at elevated temperatures.

This paper presents the greater part of the test results obtained thus far in a program of creep testing of wrought copper alloys at elevated temperatures which was initiated several years ago in the laboratories of the Chase Brass and Copper Co.

The creep literature on wrought copper alloys, while sparse as compared with that on steels, does contain several notable contributions. Among these may be mentioned the works of Clark and White (**1**, **2**, **3**),[2] Tapsell and Johnson (**4**), Tapsell, Johnson, and Clenshaw (**5**). The available data were summarized in 1937 by the British Non-Ferrous Metals Research Association (**6**) and in 1938 by The American Society of Mechanical Engineers and the American Society for Testing Materials jointly (**7**).

As the single-step tests reported here were for the most part carried on for several thousand hours, it is hoped that they will considerably amplify the existing knowledge. Most of the previously reported creep tests on copper alloys have been for distinctly shorter periods.

Materials

Materials for the test consisted of the several copper alloys listed in Table I.

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[1] Research Metallurgist, Research Assistant, and Research Physicist, respectively, Chase Brass and Copper Co., Waterbury, Conn.

[2] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 689.

IN BINDING

These were prepared in the tempers shown to permit evaluation of the effect of grain size in annealed tempers and of degree of reduction in drawn tempers. All wire was drawn in coils to the final diameter, 0.125 in., and was then mechanically straightened and cut to proper length. Material to be tested in the annealed or soft condition was annealed in these straight lengths in order that they might be subjected to a minimum of distortion before actual testing started.

It will be noted that two analyses of 70-30 brass are listed in Table I. It is believed that the slight difference in composition of the two lots is of negligible significance.

TABLE I.—COMPOSITIONS AND TEMPERS OF ALLOYS TESTED.

Alloy	Analysis by Weight, per cent	Temper
70-30 brass…………	(1) 70.45 Cu, 0.01 Fe, <0.05 Pb, 29.54 Zn^a	(*a*) Annealed: 0.200 and 0.022 mm. grain sizes (*b*) Drawn 37% and 84% (coarse grained)
	(2) 69.43 Cu, 0.01 Fe, <0.05 Pb, 30.56 Zn^a	(*a*) Annealed: 0.085 and 0.016 mm. grain sizes (*b*) Drawn 37% and 84% (fine grained)
Silicon bronze………	96.34 Cu, 2.80 Si, 0.06 Fe, 0.005 Pb, 0.80 Zn^a	(*a*) Annealed: 0.100 and 0.008 mm. grain sizes (*b*) Drawn: 37% and 84%
85-15 brass…………	84.79 Cu, 0.004 Fe, 0.002 Pb, 15.20 Zn^a	(*a*) Annealed: 0.060 mm. grain size (*b*) Drawn: 37% and 84%
Naval brass…………	59.98 Cu, 0.65 Sn, 0.015 Fe, <0.05 Pb, 39.35 Zn^a	(*a*) Annealed at 1000 F. (*b*) Drawn: 37%
Admiralty brass……	71.06 Cu, 0.92 Sn, 0.008 Fe, 0.015 Pb, 27.99 Zn^a	(*a*) Annealed: 0.055 mm. grain size (*b*) Drawn: 60%
Phosphor bronze……	94.16 Cu, 5.56 Sn, 0.24 P, 0.01 Fe	(*a*) Annealed: 0.050 mm. grain size (*b*) Drawn: 84%
70-30 copper-nickel…	69.07 Cu, 30.06 Ni, 0.80 Mn, 0.03 Fe, 0.005 Pb	(*a*) Annealed: 0.020 mm. grain size (*b*) Drawn: 84%

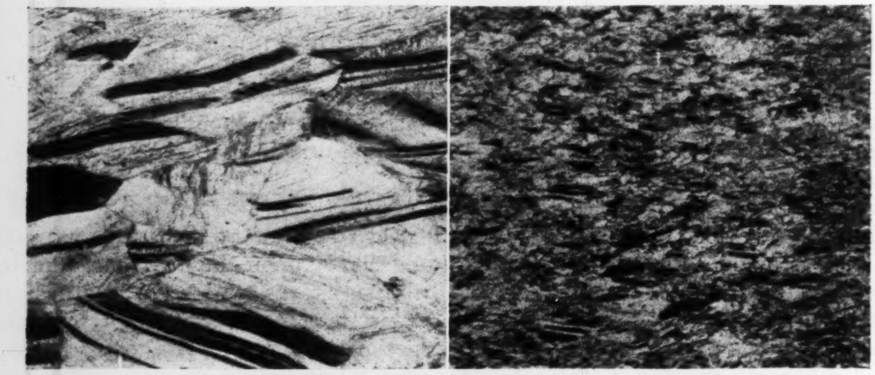
[a] By difference.

The microstructures of the 70-30 brass in the various drawn tempers are shown in Fig. 1 in order that the distinctions in grain size prior to the final cold working may be appreciated. Figure 2 shows the alpha plus beta structure of the naval brass as drawn; its structure as annealed at 1000 F. appears almost identical at a magnification of 75 diameters. The microstructures of the other alloys in the drawn condition were typical for solid-solution alloys drawn after anneals which produced moderate grain size, except for the silicon bronze as drawn 37 per cent, which was annealed to a grain size of about 0.100 mm. before the final draw.

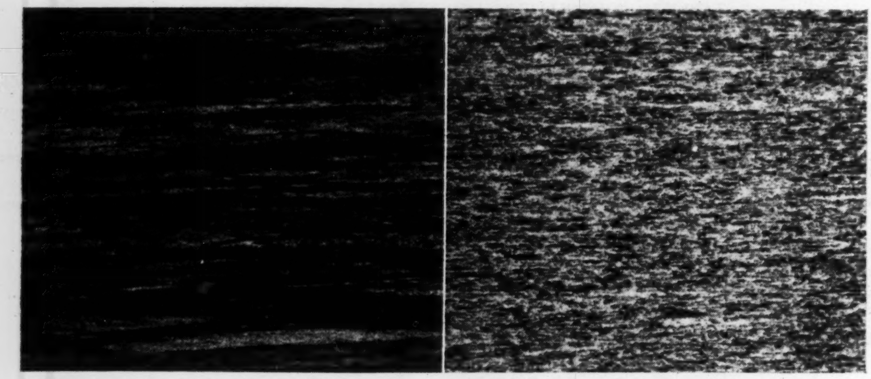
TESTING EQUIPMENT AND PROCEDURE

The creep tests were made at 300, 400, and 500 F. upon the 0.125-in. diameter wire specimens, with gage length of 10 in., in three forced convection, multiple-specimen furnaces. The respective furnaces permitted the simultaneous testing of 15, 30, and 30 specimens. As the furnaces were developed in the authors' laboratory and were of an unusual design when first built, their characteristics will be described briefly.

Each furnace consisted essentially of a sheet-iron box mounted in a frame of angle iron and lined with insulating brick to form a long rectangular chamber 6 in. wide and 20 in. high. Electric heaters and blowers were provided at each end to heat and circulate the air through the furnace chamber. In order to produce the complete and positive



(*a*) Coarse grained, drawn 37 per cent. (*b*) Fine grained, drawn 37 per cent.



(*c*) Coarse grained, drawn 84 per cent. (*d*) Fine grained, drawn 84 per cent.

FIG. 1.—Microstructures of Hard-Worked 70-30 Brass (× 75).

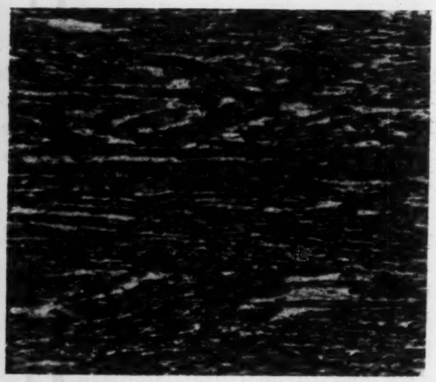


FIG. 2.—Microstructure of Naval Brass, Drawn 37 per cent, Showing Alpha and Beta Grains (× 75).

circulation required for satisfactory temperature distribution, a horizontal sheet-iron baffle extended along the entir length of the chamber and formed tw paths for the flow and counter flow of th air. The test specimens were suspende in a row along the center line of the furnace and passed through holes in th horizontal baffle. Load was applied b dead weights or by calibrated levers a required. Sponge rubber washers wer placed under the specimen support while the furnace itself was set upon rubber pads to minimize vibration effects.

Extension of the test specimens wa measured by means of a telescope fitte with a micrometer eyepiece. Spirit levels were mounted on the telescope to permit the full accuracy, ±0.0001 in., of the instrument to be obtained. The telescope was sighted through pyrex plate glass windows upon lines scratched upon two polished monel metal or stainless steel reference blocks which were attached to the wire specimen. These blocks were carefully arranged next to each other at the lower end of the 10-in. gage length, the block from the upper end being carried down by a wire which was of exactly the same material as the test wire in order to avoid differences in thermal expansion.[3] This arrangement made it possible to observe extension by determining the relative displacement of the upper and lower gage marks in one reading without disturbing the position of the telescope.

Temperature distribution and control were well within the limits set in A.S.T.M. Standard Recommended Practice for Conducting Long-Time High-Temperature Tension Tests of Metallic Materials (E 22 - 41).[4] The greatest variation in mean specimen temperature was 5 F. and the greatest variation along the 10-in. test length of a single specimen was 6 F. The maximum variations occurred in the specimens at the ends of the furnaces. Much greater uniformity existed in the other specimens. The variation in temperature with time that occurred in the control cycle was 2 F. Control of the two furnaces operated at 400 and 500 F. was accomplished by automatic potentiometer controllers, while that of the furnace at 300 F. was effected by a mercury regulator.

The room temperature physical properties of all materials were determined before and after exposure in creep tests. Specimens were examined microscopically for any indication of subsurface oxidation or deterioration and, in the case of hard-drawn tempers, of recrystallization or softening.

Creep test specimens were suspended in the furnaces for at least 24 hr. before loading. Initial extension was observed and then readings were made at suitable intervals to define the course of the creep or time-extension curve. In some of the earlier tests this initial extension upon loading was not actually determined but was calculated from stress-strain curves obtained on material at the testing temperature. Duration of the tests was of the order of 5000 hr. except when it was believed that creep rate was proceeding at a steady rate earlier, or when creep was excessive. Duplicate specimens were employed in most instances, although the most recent practice has favored the use of a greater number of stresses for single specimens of a given material.

The normal creep curves obtained were analyzed according to the McVetty type of formula (**8**):

$$\text{Creep} = e_o + vt$$

where e_o = a constant (intercept for 0 time on creep-time plot),
v = creep rate (slope or tangent to creep curve), and
t = time.

The creep rate, with a few exceptions, was taken from the second stage of the creep curves—that in which the rate of creep was constant or decreasing only slightly. Plotting of creep rate as a function of stress on logarithmic scales

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[3] The accurate determination of the creep of hard-drawn wires which recrystallize in the course of testing depends in part upon any possible effect of stress on recrystallization. The extensive tests of this investigation showed that stressed and unstressed wires of the same hard-drawn material recrystallized in a quite similar manner for given conditions of exposure at elevated temperature.

[4] 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 206.

TABLE II.—RESULTS OF CREEP TESTS.

Temper	Testing Temperature, deg. Fahr.	Stress, psi.	Total Time, hr.	Initial Extension, per cent	Total Creep, per cent	Creep		Stress for Designated Creep Rate, psi.		
						Intercept (e_o), per cent	Rate (v), per cent per 1000 hr.	0.001 per cent per 1000 hr.	0.01 per cent per 1000 hr.	0.1 per cent per 1000 hr.
70–30 **Brass**										
Annealed—0.200 mm. grain size	400	2 180	3 300	0.015	0.005	0.003	<0.001	2 400		
		2 680	3 000	0.020	0.007	0.003	0.0012			
		3 490	3 000	0.025	0.011	0.007	0.0015			
		3 580	1 680	0.028	0.017	0.013	0.0019			
		4 490	2 100	0.035	0.019	0.014	0.0028			
		5 380	3 290	0.040	0.029	0.015	0.0042			
		6 000	3 900	0.045	0.044	0.026	0.0046			
	500	1 140	2 400	0.020	0.000	0.000	<0.001	1 950	2 700	3 700
		1 950	2 400	0.025	0.005	0.003	0.0010			
		2 750	4 080	0.030	0.060	0.006	0.013^a			
		3 680	4 500	0.040	0.305	—0.112	0.093^a			
Annealed—0.085 mm. grain size	300	4 500	4 680	0.022	0.007	0.006	0.0004	9 500	16 000^b	
		10 100	5 210	0.077	0.033	0.026	0.0012			
		11 750	4 770	0.530	0.077	0.070	0.0016			
		15 100	4 770	1.69	0.242	0.21	0.0060			
	400	3 050	5 400	0.015	0.019	0.011	0.0014		7 700	
		4 000	5 060	0.020	0.028	0.019	0.0018			
		5 900	5 400	0.030	0.033	0.014	0.0034			
		8 050	5 060	0.045	0.102	0.031	0.014			
	500	1 460	4 650	0.015	0.020	0.007	0.0033	1 000	2 000	4 000
		3 020	5 000	0.030	0.155	—0.033	0.037			
		6 000	5 000	0.058	2.17	0.29	0.38			
		6 150	5 000	0.059	2.50	0.34	0.44			
Annealed—0.022 mm. grain size	400	2 790	3 300	0.008	0.044	0.016	0.0081		3 000	8 500
		4 450	1 600	0.015	0.060	0.020	0.025			
		5 690	1 600	0.019	0.085	0.025	0.036			
		8 450	3 300	0.029	0.329	0.029	0.092			
		8 580	3 300	0.030	0.400	0.035	0.11			
	500	1 125	4 080	0.013	0.102	0.014	0.021		800^b	2 200
		1 930	3 720	0.020	0.337	0.061	0.074			
		2 730	4 080	0.028	0.88	0.090	0.195			
		3 680	3 720	0.037	1.52	0.147	0.37			
Annealed—0.016 mm. grain size	300	4 500	5 210	0.017	0.028	0.016	0.0022		13 000	
		9 080	4 800	0.035	0.073	0.050	0.0046			
		15 050	4 680	0.058	0.146	0.081	0.014			
		20 100	4 800	0.075	0.495	0.240	0.053			
	400	1 980	3 240	0.015	0.029	0.011	0.0057		2 900	
		3 040	5 400	0.021	0.083	0.023	0.011			
		5 980	5 400	0.041	0.298	0.069	0.042			
	500	990	5 000	0.012	0.178	0.038	0.028		650^b	1 750
		1 480	4 650	0.017	0.318	0.033	0.061			
		3 050	2 950	0.035	1.46	0.255	0.41			
Drawn 37 per cent (coarse grained)	400	2 700	1 700	0.020	0.012	0.010	<0.001	3 300	18 000	
		8 700	5 000	0.057	0.050	0.031	0.0038			
		17 200	5 000	0.112	0.112	0.066	0.0092			
		22 600	4 300	0.150	0.190	0.12	0.016			
		26 100	5 640	0.170	0.327	0.15	0.031			
	500	1 100	5 000	0.008	0.143	0.063	0.016		900^b	
		2 750	3 000	0.018	0.47	0.130	0.115			
		5 680	1 340	0.040	0.98	—0.105	0.82^a			
Drawn 37 per cent (fine grained)	300	14 950	4 800	0.080	0.080	0.059	0.0044		24 000	44 000
		19 900	5 230	0.11	0.12	0.090	0.0064			
		29 800	4 800	0.19	0.28	0.19	0.018			
		49 550	4 680	0.37	1.53	0.47	0.22			
	400	1 000	5 400	0.006	0.024	—0.012	0.007^a			
		1 980	6 500	0.012	0.074	—0.010	0.013^a			
		3 500	6 500	0.022	0.138	—0.043	0.027^a			
		5 040	12 200	0.032	0.395	—0.124	0.042^a			
	500	620	5 000	0.006	0.18	0.07	0.021		450^b	1 150
		1 000	4 650	0.010	0.45	0.08	0.080			
		2 080	4 800	0.022	1.89	0.16	0.37			

TABLE II.—(*Continued*).

Temper	Testing Temperature, deg. Fahr.	Stress, psi.	Total Time, hr.	Initial Extension, per cent	Total Creep, per cent	Creep		Stress for Designated Creep Rate, psi.		
						Intercept (e_o), per cent	Rate (v), per cent per1000 hr.	0.001 per cent per 1000 hr.	0.01 per cent per 1000 hr.	0.1 per cent per 1000 hr.
70–30 BRASS—(*Continued*)										
Drawn 84 per cent (coarse grained)	400	710	5 000	0.005	0.024	0.024	<0.001		2 000	
		1 960	5 000	0.014	0.118	0.072	0.0093			
		2 780	3 000	0.019	0.148	0.073	0.026			
		3 690	5 000	0.025	0.36	0.105	0.052			
		4 560	1 500	0.032	0.30	0.185	0.074			
		5 880	1 500	0.035	0.43	0.195	0.16			
	500	740	3 500	0.012	0.36	0.10	0.076		300[b]	
		1 130	2 900	0.020	0.83	0.18	0.225			
		2 760	260	0.048	0.92	0.10	3.1[a]			
Drawn 84 per cent (fine grained)	300	10 000	2 230	0.086	0.062	0.055	0.0033		22 000	50 000
		19 700	5 230	0.155	0.165	0.125	0.0079			
		50 600	4 750	0.41	1.00	0.54	0.10			
		60 000	24	0.49	0.29					
	400	1 980	5 060	0.017	0.242	0.126	0.023		1 400[b]	3 500
		3 060	5 060	0.026	0.504	0.180	0.064			
		5 030	4 700	0.044	1.45	0.213	0.27			
	500	810	5 000	0.010	0.96	0.175	0.16		250[b]	700[b]
		1 480	2 620	0.015	3.0	0.212	1.07			
		4 150	170	0.050	2.28					
SILICON BRONZE										
Annealed—0.100 mm. grain size	400	5 890	5 000	0.059	0.025	0.020	0.001		8 500	
		6 010	3 290	0.060	0.020	0.017	0.001			
		7 720	3 630	0.150	0.046	0.024	0.0064			
		8 900	4 500	0.57	0.23	0.17	0.014			
		9 020	5 000	0.61	0.27	0.20	0.015			
		10 800	5 000	1.60	0.54	0.36	0.032			
	500	1 140	2 230	0.014	0.003	0.003	<0.001	3 500	5 [illegible]0	8 000
		3 040	4 460	0.036	0.009	0.004	<0.001			
		4 450	5 000	0.046	0.025	0.005	0.0040			
		6 160	5 100	0.074	0.17	0.059	0.022			
		8 100	4 630	0.149	0.80	0.26	0.12			
Annealed—0.008 mm. grain size	300	10 100	4 680	0.06	0.02	0.015	0.0009	10 000	20 000	
		19 800	4 680	0.16	0.13	0.086	0.0094			
	400	2 170	5 160	0.014	0.027	0.018	0.0018	1 600[b]	5 000	15 000[b]
		5 800	3 310	0.036	0.085	0.038	0.014			
		8 140	3 290	0.052	0.146	0.063	0.025			
		13 300	5 000	0.085	0.54	0.175	0.074			
	500	990	4 650	0.009	0.034	0.022	0.0025	700[b]	1 700	4 200
		1 950	5 100	0.018	0.131	0.044	0.017			
		4 120	5 100	0.039	0.64	0.235	0.078			
		8 030	4 130	0.076	2.61	0.320	0.55			
Drawn 37 per cent	300	29 400	4 680	0.22	0.09	0.08	0.0035		40 000	
		59 400	5 200	0.60	0.67	0.46	0.041			
	400	2 800	2 200	0.020	0.023	0.023	<0.001	4 500	24 000	
		8 800	5 230	0.062	0.059	0.046	0.0024			
		17 750	5 230	0.125	0.14	0.101	0.0070			
		22 800	4 300	0.159	0.16	0.127	0.0084			
		27 300	4 820	0.190	0.24	0.184	0.012			
	500	995	6 300	0.010	0.046	0.006	0.0062		(2 500 from abnormal curves)	7 500
		1 470	10 000	0.014	0.060	—0.006	0.0070			
		1 960	6 300	0.020	0.051	0.003	0.0076			
		3 200	5 080	0.030	0.076	0.008	0.014			
		4 970	5 700	0.044	0.200	—0.034	0.041[a]			
		8 700	5 700	0.077	0.460	—0.32	0.14[a]			
Drawn 84 per cent	300	24 750	4 500	0.17	0.09	0.07	0.0040		46 000	
		29 600	4 500	0.20	0.09	0.07	0.0049			
		38 800	4 400	0.36	0.18	0.15	0.0072			
		59 700	4 900	0.45	0.41	0.33	0.017			
		70 000	24	0.55	0.20					
	400	2 750	1 900	0.021	0.019	0.018	<0.001	4 000	24 000	
		6 100	5 000	0.046	0.048	0.041	0.0017			
		8 800	5 100	0.066	0.087	0.069	0.0030			
		18 000	5 150	0.136	0.15	0.12	0.0065			
		26 400	5 150	0.200	0.25	0.19	0.0125			
		27 150	5 150	0.207	0.24	0.18	0.0115			
	500	1 120	3 600	0.011	0.076	0.06	0.0046	650[b]	1 500	3 200
		1 950	2 400	0.018	0.154	0.10	0.023			
		2 750	3 100	0.026	0.36	0.16	0.062			
		5 900	1 200	0.056	1.34	0.17	0.97			

Continued on pp. 674 & 675

TABLE II.—(*Continued*).

Temper	Testing Temperature, deg. Fahr.	Stress, psi.	Total Time, hr.	Initial Extension, per cent	Total Creep, per cent	Creep		Stress for Designated Creep Rate, psi.		
						Intercept (e_o), per cent	Rate (v), per cent per 100 hr.	0.001 per cent per 1000 hr.	0.01 per cent per 1000 hr.	0.1 per cent per 1000 hr.
				85-15 Brass						
Annealed—0.060 mm. grain size	300	4 500	4 500	0.023	0.008	0.006	0.0007	4 900	8 000	
		9 800	4 500	0.52	0.55	0.44	0.026			
	400	2 050	5 060	0.020	0.022	0.013	0.0019		6 200	
		5 040	5 400	0.050	0.047	0.015	0.0059			
		9 120	5 060	0.56	0.94	0.56	0.077			
Drawn 37 per cent	300	9 900	4 800	0.061	0.049	0.037	0.0024		17 000	
		30 200	4 800	0.198	0.43	0.220	0.043			
	400	7 900	5 400	0.043	0.088	0.054	0.0066		9 200	
		8 100	5 400	0.044	0.091	0.054	0.0069			
		15 650	4 420	0.092	0.33	0.151	0.041			
		25 950	2 300	0.173	2.37	—0.080	1.05^a			
	500	980	10 370	0.009	0.17	0.055	0.011			
		3 050	10 000	0.027	0.94	0.073	0.086			
		6 050	4 800	0.054	1.06	—0.70	0.37^a			
Drawn 84 per cent	300	9 960	4 400	0.063	0.033	0.029	0.0007		37 000	
		39 700	4 500	0.290	0.155	0.108	0.011			
	400	1 040	5 160	0.005	0.035	0.029	0.0011		2 600	
		3 190	5 000	0.017	0.146	0.061	0.017			
		5 400	5 160	0.031	0.43	0.11	0.063			
		6 040	5 000	0.035	0.47	0.12	0.069			
	500	960	5 300	0.010	0.26	0.080	0.034			
		1 940	2 950	0.018	0.66	0.104	0.19			
		2 990	3 650	0.025	2.69	—0.045	0.76^a			
				Naval Brass						
Annealed—at 1000 F.	300	4 600	4 750	0.030	0.027	0.015	0.0026		10 500	
		10 050	4 500	0.067	0.065	0.022	0.0096			
		15 100	4 500	0.101	0.127	0.048	0.017			
	400	1 150	5 000	0.007	0.038	0.019	0.0037		1 900	5 000
		1 575	5 000	0.010	0.065	0.027	0.0075			
		2 800	3 630	0.018	0.105	0.025	0.022			
		4 180	4 730	0.027	0.36	0.080	0.061			
		5 950	4 820	0.040	0.96	0.035	0.19			
		12 900	550	0.090	2.0					
	500	730	2 180	0.003	0.25	0.059	0.084		250^b	800
		1 980	1 080	0.012	0.81	0.032	0.71			
Drawn 37 per cent	300	4 580	4 750	0.036	0.084	0.059	0.0052		9 000	
		15 050	4 900	0.117	0.38	0.305	0.015			
	400	1 150	5 000	0.012	0.056	0.033	0.0048		3 000	9 000
		1 570	5 000	0.016	0.070	0.040	0.0059			
		2 820	5 160	0.027	0.12	0.079	0.0090			
		5 920	4 820	0.056	0.37	0.220	0.031			
		13 700	1 220	0.128	1.2	0.53	0.48			
	500	320	1 440	0.005	0.09					
		740	2 160	0.011	0.48					
		1 160	1 080	0.016	0.48					
		1 980	900	0.029	1.67					
				Admiralty Brass						
Annealed—0.055 mm. grain size	300	4 540	4 500	0.016	0.013	0.013	<0.001			
		12 050	3 380	0.062	0.028	0.023	0.0014			
		15 150	4 400	0.081	0.053	0.042	0.0028			
		19 850	0	>2.5						
	500	980	4 980	0.003	0.023	0.003	0.0037		2 200	5 000
		1 970	4 300	0.007	0.065	0.031	0.0080			
		3 000	4 980	0.012	0.137	0.034	0.021			
		4 500	4 300	0.021	0.285	0.019	0.062^a			
		6 150	6 300	0.033	1.495	—0.350	0.295^a			
Drawn 60 per cent	500	990	2 950	0.010	0.415	0.090	0.11			900^b
		2 950	2 600	0.031	2.570	0.340	0.86			

TABLE II—(*Concluded*).

Temper	Testing Temperature, deg. Fahr.	Stress, psi.	Total Time, hr.	Initial Extension, per cent	Total Creep, per cent	Creep		Stress for Desginated Creep Rate, psi.		
						Intercept (e_o), per cent	Rate (v), per cent per 1000 hr.	0.001 per cent per 1000 hr.	0.01 per cent per 1000 hr.	0.1 per cent per 1000 hr.
PHOSPHOR BRONZE										
Annealed—0.050 mm. grain size	300	4 530	5 620	0.023	0.005	0.003	0.0003	11 000		
		10 000	5 620	0.058	0.014	0.009	0.0008			
		15 100	5 620	0.115	0.027	0.016	0.0019			
		19 850	—	>2.1	—					
	400	1 960	4 100	0.010	0.009	0.008	0.0003			
		4 000	5 400	0.017	0.043	0.027	0.0027			
	500	1 000	5 000	0.008	0.023	0.005	0.0036			
		4 970	5 800	0.045	0.171	0.065	0.018			
Drawn 84 per cent	300	4 580	5 620	0.031	0.014	0.009	0.0009	5 000	32 000[b]	
		14 950	5 620	0.105	0.065	0.047	0.0032			
		19 800	5 620	0.138	0.097	0.075	0.0037			
		29 800	5 620	0.210	0.157	0.112	0.0080			
	400	5 870	5 060	0.040	0.100	0.071	0.0061		7 500	
		10 400	4 100	0.077	0.211	0.131	0.020			
	500	990	6 300	0.010	0.670	0.280	0.062		2 700	
		3 020	1 780	0.034	2.68	—0.034	1.53[a]			
		4 960	830	0.055	2.63					
70–30 COPPER NICKEL										
Annealed—0.020 mm. grain size	300	4 550	2 600	0.023	0.010	0.010	<0.0001	24 000	35 000[b]	
		15 100	2 600	0.087	0.022	0.022	<0.0001			
		19 900	4 400	0.86	0.024	0.022	0.0004			
		28 450	3 400	3.20	0.026	0.019	0.0023			
	400	4 980	2 880	0.022	0.004	0.004	<0.0001			
		15 950	4 100	0.160	0.007	0.006	0.0002			
	500	4 980	2 780	0.030	0.001	0.001	<0.0001	16 000	30 000	
		10 250	5 000	0.062	0.013	0.013	<0.0005			
		15 650	3 260	0.29	0.011	0.008	0.0009			
		21 150	5 000	1.45	0.048	0.030	0.0037			
		26 500	3 260	2.72	0.045	0.028	0.0054			
Drawn 84 per cent	300	19 900	3 600	0.090	0.018	0.018	<0.0001			
	500	4 960	2 760	0.022	0.014	0.012	0.0007	8 000		
		10 100	4 980	0.045	0.029	0.024	0.0011			
		20 800	4 800	0.093	0.033	0.023	0.0021			
		40 800	4 800	0.182	0.082	0.067	0.0035			

[a] Rate taken from third stage of creep.
[b] By extrapolation.

provided means of comparison of test results.

DISCUSSION OF RESULTS

The results of the creep tests are shown in detail in Table II. The tensile properties of the test materials at room temperature before and after exposure in the creep tests, as well as notes on changes in microstructure, are listed in Table III. For the sake of brevity, properties after exposure at the elevated temperatures are not given in those instances where no change was observed. The results obtained for each material have been represented graphically and are included in the following discussion.

Figure 3 is included to illustrate the general character of the creep or time-extension curves most frequently obtained in the investigation, for all the individual curves cannot be reported here.

TABLE III.—ROOM TEMPERATURE PROPERTIES BEFORE AND AFTER CREEP TESTS.

Temper	Creep Testing			Yield Strength (½ per cent elongation), psi.	Tensile Strength, psi.	Elongation in 2 in., per cent	Microstructure
	Temperature, deg. Fahr.	Stress, psi.	Time, hr.				
70–30 BRASS							
Annealed: 0.200 mm. grain size	...	....	....	8 500	42 500	55	alpha grains
	500	2 800	4 560	....	36 000	38	no change
		0	4 560	....	42 000	52	no change
		3 690	5 850	....	19 000	12	no change
		0	5 850	....	42 000	54	no change
0.085 mm. grain size	...	....	....	11 500	48 500	57	alpha grains
0.022 mm. grain size	...	....	....	19 000	52 000	49	alpha grains
0.016 mm. grain size	...	....	....	22 500	59 500	42	alpha grains
Drawn 37 per cent (coarse grained)	...	....	....	....	85 000	7	hard drawn
	400	2 780	2 200	....	83 000	....	as drawn
		0	2 200	....	82 000	....	as drawn
		17 900	5 280	....	81 000	7	partially recrystallized
		0	5 280	....	80 500	10	partially recrystallized
		26 800	5 780	....	54 000	1	partially recrystallized
		0	5 780	....	81 000	10	partially recrystallized
	500	1 080	340	....	56 000	37	partially recrystallized
		5 800	1 700	....	52 500	46	partially recrystallized
		0	1 700	....	52 500	47	partially recrystallized
		1 060	5 000	....	53 000	55	completely recrystallized to 0.028 mm. grain size, not uniform
Drawn 37 per cent (fine grained)	...	....	....	62 000	86 000	10	hard drawn
	300	49 400	5 040	....	89 000	6.5	as drawn
	300	0	5 040	....	88 500	6.5	as drawn
	400	1 000	5 400	....	69 000	26	recrystallization beginning
	400	5 040	12 300	....	60 500	38	recrystallization substantially complete
	500	2 080	4 970	....	55 500	43	completely recrystallized to 0.014 mm. grain size
Drawn 84 per cent (coarse grained)	...	....	....	....	124 500	5.5	hard drawn
	400	1 060	770	....	69 000	32	completely recrystallized
	400	8 770	5 000	....	67 000	34	completely recrystallized (0.010 mm.)
	500	17 800	16	....	67 500	30	completely recrystallized
	500	1 100	2 900	....	62 000	37	completely recrystallized
Drawn 84 per cent (fine grained)	...	....	....	65 000	120 000	4.5	hard drawn
	300	50 600	4 870	....	130 500	4	as drawn
	300	0	4 870	....	127 500	4	as drawn
	400	21 100	500	....	71 500	28	completely recrystallized
	400	5 030	5 050	....	68 500	34	completely recrystallized (<0.005 mm.)
	500	1 480	3 400	....	63 500	35	completely recrystallized (0.008 mm.)
SILICON BRONZE							
Annealed—0.100 mm. grain size	...	....	....	8 000	49 500	65	alpha grains
	400	9 020	5 000		49 500	65	no change[a]
		0	5 000		49 500	63	
	500	15 100	500		50 500	54	no change[a]
		0	500		49 500	67	
		6 160	5 100		47 500	51	
		0	5 100		49 000	62	
Annealed—0.008 mm. grain size	...	....	....	25 000	61 500	61	alpha grains
	400	2 170	9 050		57 500	49	no change[a]
		0	9 050		57 500	46	
		13 300	5 200		60 000	47	
		0	5 200		60 000	49	
	500	8 030	4 900		58 000	44	no change[a]
		0	4 900		59 000	48	
Drawn 37 per cent	...	....	....	58 000	90 500	9	hard drawn
	400	8 900	5 050		87 000	10	as drawn[a]
		27 250	5 000		88 000	10.5	
	500	2 780	1 770		89 000	10	as drawn[a]
		0	1 770		89 000	10	
		8 700	5 690		66 000	26	partially recrystallized[a]
		8 700	5 690		67 500	34	

TABLE III.—(*Concluded*).

Temper	Creep Testing			Yield Strength (½ per cent elongation), psi.	Tensile Strength, psi.	Elongation in 2 in., per cent	Microstructure
	Temperature, deg. Fahr.	Stress, psi.	Time, hr.				
SILICON BRONZE—*continued*							
Drawn 84 per cent	...	....	....	68 500	143 000	5.5	hard drawn
	300	59 700	4 950		148 000	3.5	as drawn
		0	4 950		147 000	4	
	400	2 770	2 370		141 000	3.5	as drawn[a]
		27 150	9 050		134 000	3.5	
	500	1 120	3 600		75 000	36	completely recrystallized[a]
		0	3 600		75 000	36	
85–15 BRASS							
Annealed—0.060 mm. grain size	...	....	....	9 000	41 000	47	alpha grains
Drawn 37 per cent	...	....	....	59 000	67 500	10.5	hard drawn
	400	15 650	4 460		67 000	13	as drawn
		0	4 460		67 000	12	as drawn
	500	3 050	1 320		56 500	20	no apparent recrystallization
		0	1 320		58 500	21	no apparent recrystallization
		1 000	8 600		45 000	42	completely recrystallized
		0	8 600		44 000	41	completely recrystallized
Drawn 84 per cent	...	....	....	67 000	96 500	7	as drawn
	400	6 040	5 000		52 500	37	almost completely recrystallized
		0	5 000		53 500	35	almost completely recrystallized
		1 040	8 980		51 500	34.5	completely recrystallized
		0	8 980		51 500	39	completely recrystallized
	500	2 990	3 900		47 000	32	completely recrystallized
		0	3 900		49 500	40	completely recrystallized
NAVAL BRASS							
Annealed—at 1000 F.	...	....	....	30 500	66 000	40	alpha + beta
	300	4 600	4 870		65 500	37.5	alpha + beta + third phase
	400	5 800	5 000		67 000	32	alpha + beta + third phase
		0	5 000		66 000	32	alpha + beta + third phase
	500	1 950	1 460		66 500	35.5	alpha + beta + third phase
		0	1 460		66 000	35.5	alpha + beta + third phase
Drawn 37 per cent	...	....	....	66 000	95 000	10	alpha + beta—drawn
	300	15 050	4 950		95 000	13.5	as drawn + third phase
		0	4 950		98 000	11	as drawn + third phase
	400	5 800	5 000		92 000	18.5	partial recrystallization + third phase
		0	5 000		90 000	16.5	partial recrystallization + third phase
	500	1 900	720		77 500	28	recrystallized + third phase
		0	720		78 500	29	recrystallized + third phase
ADMIRALTY BRASS							
Annealed—0.055 mm. grain size	...	....	....	15 500	52 000	60	alpha grains
	300	15 150	4 740	....	51 000	57.5	no change
		0	4 740		51 000	58	no change
Drawn 60 per cent	...	....	....	71 000	109 000	6	hard drawn
PHOSPHOR BRONZE							
Annealed—0.050 mm. grain size	...	....	....	20 000	52 000	66	alpha grains
Drawn 84 per cent	...	....	....	77 500	144 000	3.5	hard drawn
	300	19 800	6 120		138 000	5	as drawn
	400	10 400	4 270		121 000	8	as drawn
		0	4 270		122 000	5	as drawn
	500	3 020	2 200		70 000	35	recrystallization essentially complete
70–30 COPPER NICKEL							
Annealed—0.020 mm. grain size	...	....	....	21 000	59 000	40	alpha grains
	500	4 980	2 780		59 500	38	no change
		0	2 780		59 500	37.5	no change
Drawn 84 per cent	...	....	....	80 000	90 500	9	hard drawn
	500	4 960	2 780		92 000	10	as drawn
		0	2 780		93 000	10	as drawn

---

[a] Intergranular oxidation at surface.

70-30 Brass:

The relationships between stress and creep rate for 70-30 brass at 300, 400, and 500 F. are plotted logarithmically in Fig. 4. Except as will be discussed hereinafter, the rates were determined in the second stage of creep, that stage of constant or only slightly decreasing rate. It is quite obvious that, as expected, the resistance to creep for the various tempers decreases as temperature increases.

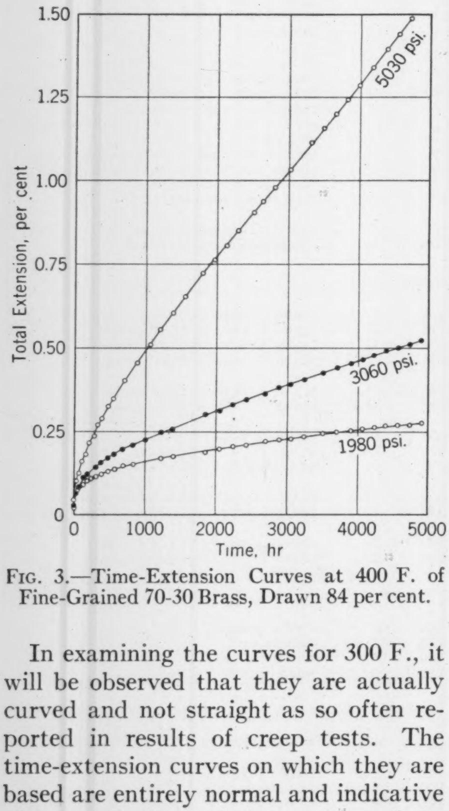

FIG. 3.—Time-Extension Curves at 400 F. of Fine-Grained 70-30 Brass, Drawn 84 per cent.

In examining the curves for 300 F., it will be observed that they are actually curved and not straight as so often reported in results of creep tests. The time-extension curves on which they are based are entirely normal and indicative of proper testing technique. Apart from this, it is evident that the load-carrying capacity of the drawn materials is greater than for the annealed materials, although for lower stresses annealed material of rather large grain size is superior to drawn material. While there is little difference in the curves for the materials drawn 37 and 84 per cent, the greater strength of the latter is indicated at the highest stress, approximately 50,000 psi. The annealed material with a grain size of 0.085 mm. is superior to that with a grain size of 0.016 mm. for moderate and low stresses. As the curves for the two are beginning to converge at the higher stresses, it might be that the relationship between them would eventually be reversed. This convergence may be associated with the yield and tensile strengths of the materials; the two highest stresses used with material of 0.085 mm. grain size are above its yield strength as indicated by the initial extension in the creep tests.

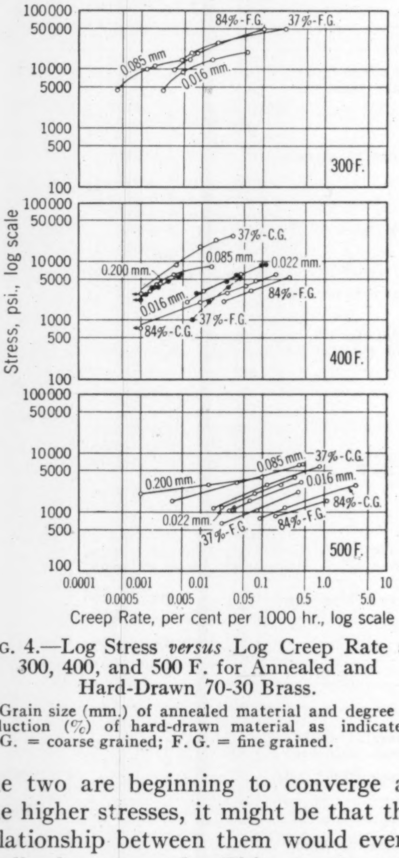

FIG. 4.—Log Stress *versus* Log Creep Rate at 300, 400, and 500 F. for Annealed and Hard-Drawn 70-30 Brass.

Grain size (mm.) of annealed material and degree of reduction (%) of hard-drawn material as indicated. C. G. = coarse grained; F. G. = fine grained.

The log-log curves for 400 F. are

essentially linear and parallel with but two exceptions. The first exception is for fine-grained material as drawn 37 per cent. In this case the test results as plotted are abnormal and should not be compared with the others, for the rates involved are taken from the third stage of creep. The time-extension or creep curves for the material at this temperature appear to have entered the third stage rather early (Fig. 5). The slopes or creep rates at the inflection points are extremely low and are not applicable over any extended period of time. The only practical value from this group of tests is therefore to be obtained from the third stage of creep. The abnormality is probably due to recrystallization and will be discussed later in the paper.

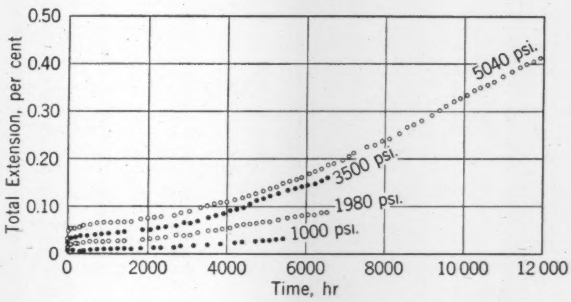

Fig. 5.—Time-Extension Curves at 400 F. of Fine-Grained 70-30 Brass, Drawn 37 per cent.

The second exception with regard to character of log-log curves at 400 F. is the curve for annealed material with 0.085 mm. grain size which lies above the 0.200-mm. grain size curve. It is indicated that it would lie below the 0.200-mm. grain size curve for stresses less than about 3000 psi. It is not known whether these two curves would cross again at some higher stress or whether the 0.200-mm. grain size curve would bend off to the right and remain below the 0.085-mm. grain size curve. The curve for the larger grain size might have been expected to lie above the curve for the smaller grain size at all times. Actually there is little quantitative difference between the two.

In this group of curves for 400 F., that for the coarse-grained material as drawn 37 per cent lies well above the others. This material is nominally capable of carrying stresses far beyond the range of the other materials. The curves for the annealed materials lie below substantially in the order of decreasing grain sizes. The two materials drawn 84 per cent lie below these, and the curve for the fine-grained material is the lower of these two.

The log-log curves for 500 F. are essentially straight and parallel with the exception of that for material with 0.200-mm. grain size. The flatness of this curve is due to the fact that the points for 2750 and 3680 psi. are from the third stage of creep. If it had been plotted from rates in the second stage of creep, the slope of the log-log curve would have been somewhat steeper and would not have intersected the 0.085-mm. grain size curve. However, the second stage was virtually nonexistent and the practical value of the test is to be obtained from the third stage. Apart from this one exception, the parallelism of this group of log-log curves is excellent and the positions of the individual curves are readily rationalized. They lie in series in the order of final grain size.

Some unusual creep curves were obtained for the annealed temper, 0.085 mm. grain size, at 500 F. with stresses of 3020, 6000, and 6150 psi. (Fig. 6). There is essentially no region of initially decreasing creep rate in these; the rates first increase and then, for the two highest stresses, decrease. It seems likely that the rate for the stress of 3020 psi. would decrease after a longer time. The shape of these curves is similar to that of creep curves described by one of the authors (9) for single crystals of 70-30 brass.

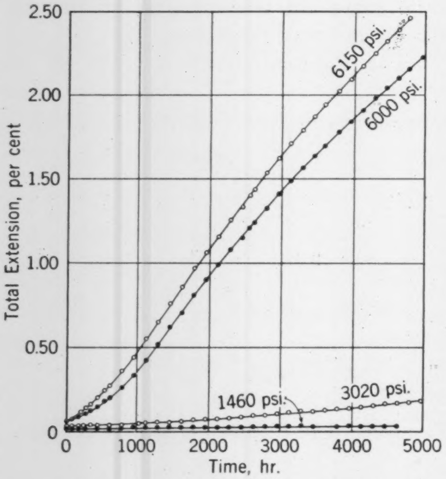

FIG. 6.—Time-Extension Curves at 500 F. of Annealed 70-30 Brass, 0.085 mm. Grain Size.

No change in the microstructure of annealed tempers of this alloy is observed after any of the creep tests. None of the drawn wires show any indication of recrystallization at 300 F., but they do undergo the slight increase in strength associated with low-temperature stress-relieving anneals.

The creep behavior of several of the drawn materials at 400 and 500 F. is associated with their recrystallization characteristics. At 400 F., wire drawn 84 per cent becomes completely recrystallized in the course of the tests and thereafter behaves as very fine-grained annealed metal. The initially coarse-grained wire recrystallizes to a slightly larger grain size than does the initially fine-grained wire. Recrystallization is presumably complete very early in the test (10), probably before the loads are applied. The alloy as drawn 37 per cent with fine grain recrystallizes slowly at this temperature. The time at which its recrystallization may be expected to begin (10) corresponds well with the inflection points of the creep curves (Fig. 5), after which creep accelerates. As drawn 37 per cent with coarse grain, the alloy was observed to begin to recrystallize sometime after 2200 hr., a time in agreement with published work (10), but recrystallization did not proceed to completion. The creep in this case did not accelerate at any time during the tests.

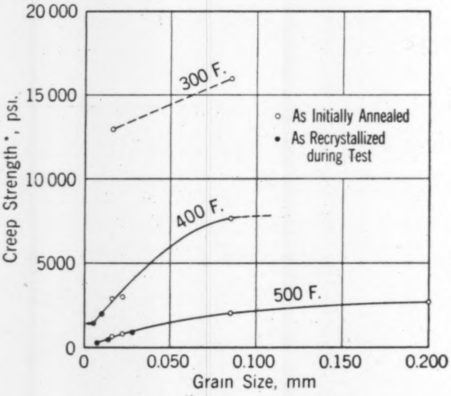

FIG. 7.—Creep Strength,* Grain Size Relationship for 70-30 Brass.

* That stress which produces the creep rate of 0.01 per cent per 1000 hr.

At 500 F. all the drawn materials recrystallized completely. That as drawn 37 per cent with coarse grain was only partially recrystallized after 1700 hr., but was completely so after 5000 hr. Recrystallization of the other drawn wires was undoubtedly complete before load was applied.

By carrying out the questionable steps of extrapolating several stress-creep rate curves back to a rate of 0.01 per cent per 1000 hr. in order to obtain values for an arbitrary creep strength and by using data upon drawn material which recrystallized at the testing temperatures, as well as data upon originally annealed metal, it is possible to arrive at the relationship between grain size and creep strength plotted in Fig. 7. While these curves would admittedly be benefited by data on material of intermediate grain size, there are indications of a relationship existing at least at 400 and 500 F.

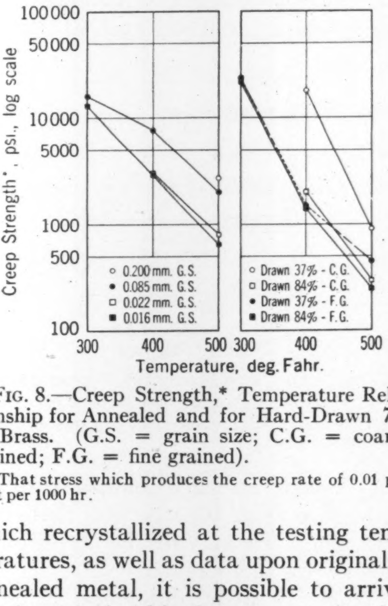

FIG. 8.—Creep Strength,* Temperature Relationship for Annealed and for Hard-Drawn 70-30 Brass. (G.S. = grain size; C.G. = coarse grained; F.G. = fine grained).

* That stress which produces the creep rate of 0.01 per cent per 1000 hr.

The same values of creep strength facilitate evaluation of the effect of temperature on comparative creep resistance of the alloy in the various conditions as plotted in Fig. 8.

Embrittlement of the 70–30 brass was encountered in two general conditions. The coarse-grained wire, drawn 37 per cent, suffered considerable loss in strength and ductility after having been stressed at 26,800 psi. at 400 F. Intercrystalline parting could not be found in the microstructure although such a condition was expected. Embrittlement had not developed at lower stresses, but it is reasonable to believe that it might have after a longer time. This is a point to consider before placing too much credence in the high creep resistance of this temper at 400 F. The annealed wire with grain size of 0.200 mm. was embrittled at all stresses used at 500 F. Its loss of strength and ductility increased with applied stress. There was, of course, no embrittlement of unstressed wires.

From the preceding discussion it is evident that the original temper of this alloy is a very important factor in determining its creep characteristics. At 300, 400, and 500 F. creep resistance of annealed material for feasible stresses increases as grain size increases within the range of normally encountered grain sizes. The grain size effect is similarly noticeable in drawn materials. In addition, the degree of cold working in hard materials is important, for it is intimately associated with recrystallization at 400 and 500 F. Coarse-grained metal may show nominally high creep strength for conditions under which it is becoming embrittled by intergranular separation and hence may be in danger of sudden failure.

The correlation of creep properties at these temperatures is partly at variance with the findings of Clark and White (2), who reported that 400 F. was below and 600 F. was above the equicohesive temperature or lowest possible recrystallization temperature of 70–30 brass and that coarse-grained material was superior to fine-grained material at temperatures above 400 F., while the reverse was true at 400 F. or lower.

It is known from the results of the present tests and also from the work of Maddigan and Blank (10), whose long-time annealing experiments were performed in conjunction with these creep tests, that the lowest possible recrystallization temperature of 70–30 brass must lie below 400 F. The present results indicate that it may be below 300 F. if the relative positions of the stress-creep rate curves for fine- and coarse-grained annealed materials are a criterion. It can be said that the creep strengths of fine- and coarse-grained 70–30 brass are approaching each other at 300 F. and that the finer grained metal may have the greater creep resistance at a somewhat lower temperature. It would at least have a greater load-carrying capacity without appreciable deformation at lower temperatures by virtue of its higher yield strength.

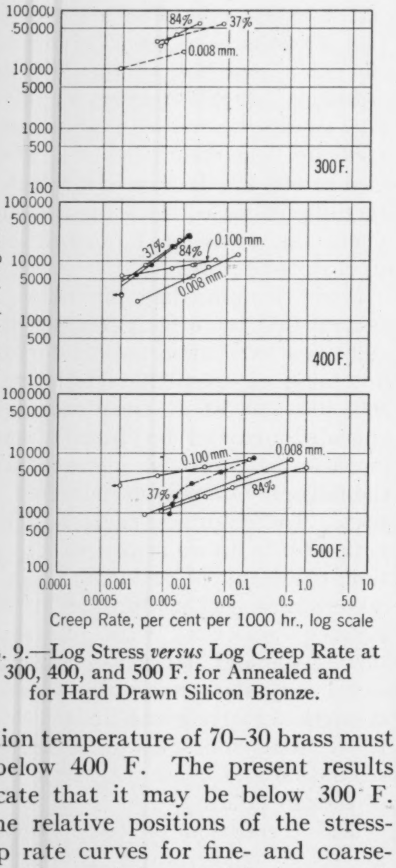

FIG. 9.—Log Stress *versus* Log Creep Rate at 300, 400, and 500 F. for Annealed and for Hard Drawn Silicon Bronze.

Silicon Bronze:

The stress, creep rate, and temperature relationships for the silicon bronze are summarized in the log-log plot of Fig. 9 and in Fig. 10.

At 400 and 500 F., the annealed material with grain size of 0.100 mm. has higher creep strength than that with grain size of 0.008 mm. Although there are no data for 0.100 mm. at 300 F., the creep strength-temperature relationship (Fig. 10) indicates that 0.008 mm. grain size may be the better of the two at this temperature.

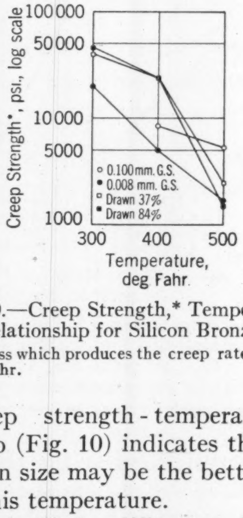

FIG. 10.—Creep Strength,* Temperature Relationship for Silicon Bronze.

* That stress which produces the creep rate of 0.01 per cent per 1000 hr.

There is little difference in creep strength of the silicon bronze as drawn 37 and 84 per cent at 400 F. At 300 F., the stress-creep rate curves intersect as the harder drawn temper becomes superior for high stresses in a manner similar to that for drawn 70–30 brass at the same temperature. At 500 F., the originally hardest material, drawn 84 per cent, has the least creep strength of all the tempers. At 300 and 400 F., the alloy was better as drawn than as annealed, but recrystallization effects lowered the relative positions of the drawn tempers at 500 F.

Attention is called to the curve for the alloy, drawn 37 per cent, at 500 F. in Fig. 9. The rates plotted were taken from the time-extension curves shown in Fig. 11. The erratic and abnormal effects to be found in the latter curves are probably due to instability during recrystallization. The material at high stress has definitely entered the third stage of creep, that of increasing rate. At the lower stresses it does the same, but later enters a stage of decreasing rate. These effects make it difficult to

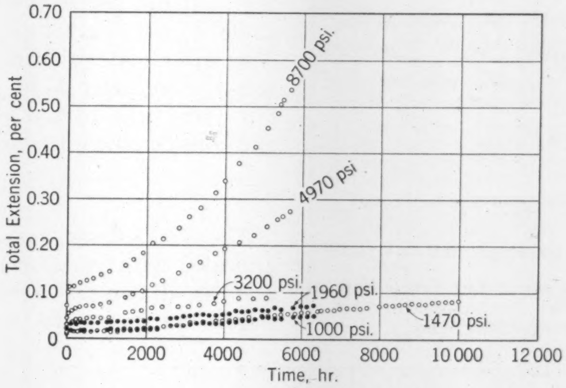

FIG. 11.—Time-Extension Curves at 500 F. of Silicon Bronze, Drawn 37 per cent.

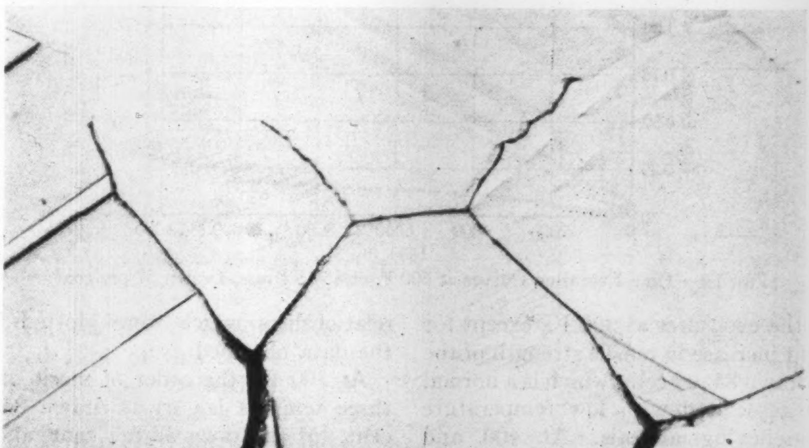

FIG. 12.—Grain Boundary Attack at 500 F. in Annealed Silicon Bronze, 0.100 mm. Grain Size (× 500).

select rates which reasonably describe the behavior of the material.

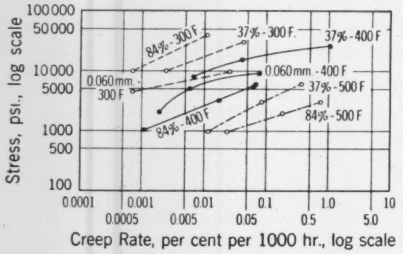

FIG. 13.—Log Stress *versus* Log Creep Rate at 300, 400, and 500 F. for Annealed and for Hard-Drawn 85-15 Brass.

No changes in structure or properties for any of the tempers were observed after the exposures at 300 F., except for a slight increase in tensile strength of the wire drawn 84 per cent, which is a normal effect associated with low temperature stress-relieving anneals. At 400 and 500 F. there occurred a slight intergranular deterioration at the metal surface, presumably due to oxidation, the depth of penetration being as much as 0.005 in. after 5000 hr. at 500 F. There was no noticeable difference in the depth of penetration in stressed and unstressed wires. However, the oxidized grain boundaries in stressed samples tended to open (Fig. 12). Some loss of tensile strength was produced by this phenomenon.

The wire drawn 84 per cent was found to have recrystallized to a very small grain size at 500 F., which accounted for its low creep resistance at this temperature. It did not recrystallize or soften at 400 F., nor did that drawn 37 per cent. The latter material had not started to recrystallize after 1770 hr. at 500 F., but there was partial recrystallization and considerable softening after 5000 hr.

85–15 Brass:

The 85–15 brass was investigated as annealed with grain size of 0.060 mm. and as drawn 37 and 84 per cent. Figure 13 shows the stress-creep rate relationships which were plotted from the data obtained.

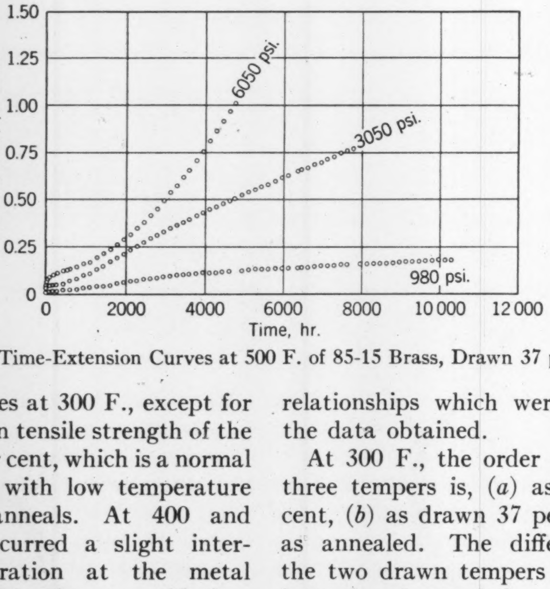

FIG. 14.—Time-Extension Curves at 500 F. of 85-15 Brass, Drawn 37 per cent.

At 300 F., the order of merit of the three tempers is, (*a*) as drawn 84 per cent, (*b*) as drawn 37 per cent, and (*c*) as annealed. The difference between the two drawn tempers is marked. It is to be observed from the value for initial extension that the higher of the two stresses for the annealed material was above the yield strength at this temperature.

The 37 per cent draw proved to be the most resistant temper at 400 F., while the 84 per cent draw was the least resistant. The rate shown for the 37 per cent draw at a stress of 25,950 psi.

was taken from the third stage of the creep curve and represents the practical interpretation of the curve. The first of the two specimens at this stress fractured in 2280 hr., after creeping about 2.7 per cent. The fracture was intercrystalline and there was much intercrystalline parting within the body of the specimen. The second specimen broke in 2880 hr.

The 37 per cent draw was also superior to the 84 per cent draw at 500 F. No data had been obtained on the annealed alloy at this temperature.

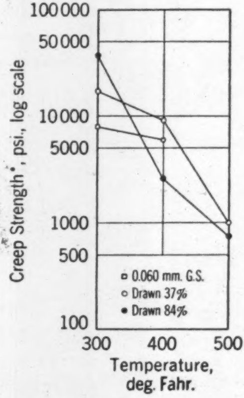

FIG. 15.—Creep Strength,* Temperature Relationship for 85-15 Brass.

* That stress which produces the creep rate of 0.01 per cent per 1000 hr.

For stresses of 980 and 3050 psi. at 500 F., the creep rates of the alloy, drawn 37 per cent, increased initially, then decreased and became substantially constant (Fig. 14). As stressed at 6050 psi. the course of creep was normal; the third stage of creep soon developed and was maintained for the duration of the test. These phenomena are quite similar to those for 70–30 brass, drawn 37 per cent, at 400 F., and to those for the silicon bronze, drawn 37 per cent, at 500 F. The underlying cause, recrystallization and softening, is the same.

The relations between creep strength and temperature for the three tempers of this alloy are shown in Fig. 15.

Except for the specimens finished with 37 per cent draw and tested under stress of 25,950 psi. at 400 F., as already described, there was no significant change in structure or tensile properties of the alloy as originally annealed or as drawn 37 per cent after the tests at 300 and 400 F. After 1320 hr. at 500 F., the material drawn 37 per cent had softened appreciably although there was no apparent recrystallization. After 8600 hr., recrystallization and softening were complete. Recrystallization in the

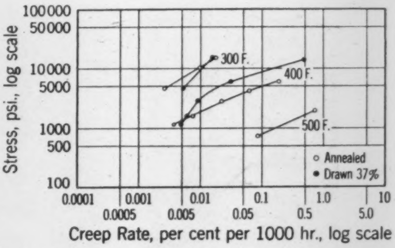

FIG. 16.—Log Stress *versus* Log Creep Rate at 300, 400, and 500 F. for Annealed and for Hard-Drawn Naval Brass.

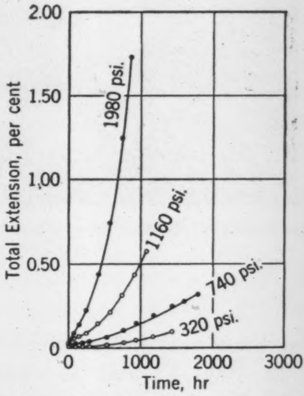

FIG. 17.—Time-Extension Curves at 500 F. of Naval Brass, Drawn 37 per cent.

alloy as drawn 84 per cent was almost complete after 5000 hr. at 400 F., and was complete after 8980 hr. At 500 F., recrystallization for this temper was definitely complete before 3900 hr. The recrystallization characteristics of drawn materials again account for their relative creep resistance with respect to each other.

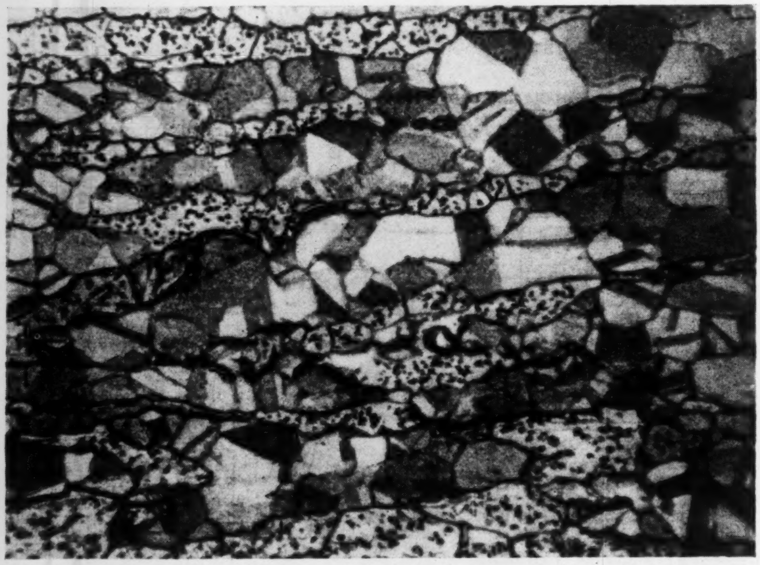

FIG. 18.—Annealed Naval Brass Showing Alpha, Beta, and Gamma Phases After Exposure in Creep Test at 400 F. for 5000 hr. (× 500).

Naval Brass:

Naval brass was investigated as annealed at 1000 F. and as drawn 37 per cent. The stress-creep rate curves for the two tempers intersect at both 300 and 400 F. (Fig. 16). That for the drawn temper at 500 F. is not shown as the creep rate was accelerating for all stresses (Fig. 17).

There was no significant change in properties of either temper after testing at 300 F., except for slight changes in elongation. After exposure at 400 and 500 F., the tensile strength of the annealed material was unchanged, but its elongation in the tension test was less than formerly. The drawn material was partially recrystallized at 400 F., and tensile strength and elongation were accordingly affected. At 500 F., there was further softening and recrystallization appeared complete.

With regard to structure, particles of a third phase, presumably gamma, were found to have developed in the original alpha plus beta structures at all the temperatures, 300, 400, and 500 F. (Fig. 18). The gamma phase is due to the presence of tin and is stable in the alloy at relatively low temperatures (11). Its absence in naval brass as ordinarily produced is due to the greater solubility of tin at commercial annealing or hot-working temperatures, where only alpha and beta are stable.

Admiralty Brass:

The creep data obtained on the annealed and the drawn Admiralty brass are summarized in Fig. 19. The alloy as drawn 60 per cent has obviously recrystallized at 500 F. and is behaving as a fine-grained, annealed material. Of the creep curves from which these creep rates were taken, those for the annealed material at 500 F. pass into the third stage between 500 and 1000 hr. for stresses of 4500 and 6150 psi. The rates for these stresses were taken from the third stage.

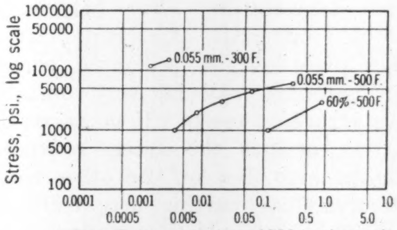

FIG. 19.—Log Stress *versus* Log Creep Rate at 300 and 500 F. for Annealed and for Hard-Drawn Admiralty Brass.

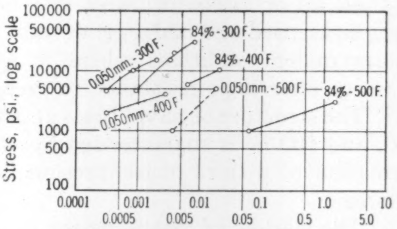

FIG. 20.—Log Stress *versus* Log Creep Rate at 300, 400, and 500 F. for Annealed and for Hard-Drawn Phosphor Bronze.

Phosphor Bronze:

The stress - creep rate data obtained for the annealed and the drawn phosphor bronze have been plotted in Fig. 20. The creep rate of the annealed material at 300 F. was very low, lower than for the drawn temper, for stresses of 15,000 psi. or less. When stressed at 19,850 psi. at 300 F., the immediate extension was greater than 2 per cent and no creep data were obtained. Comparison of the two tempers at 400 F. is difficult because of the lack of sufficient data. At 500 F., the annealed condition is easily the better of the two.

The structure and tensile properties of the annealed bronze were essentially unaffected at 300, 400, and 500 F. The drawn alloy was partly softened after 4270 hr. at 400 F. although there was no indication of recrystallization in the microstructure. At 500 F., recrystallization was practically complete after 2200 hr.

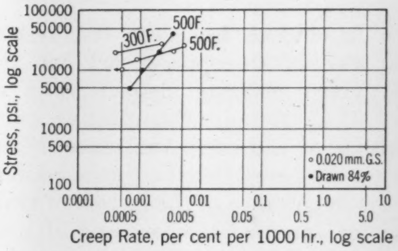

FIG. 21.—Log Stress *versus* Log Creep Rate at 300 and 500 F. for Annealed and for Hard-Drawn 70-30 Copper-Nickel.

70-30 Copper-Nickel:

The creep characteristics of the 70-30 copper-nickel alloy are shown in the stress - creep rate curves of Fig. 21. The creep rate of the alloy as annealed was almost negligible at 300 F. for stresses of 19,900 psi. or less; the initial extension indicated that 19,900 psi. exceeded the yield strength of the material under these conditions. At 500 F., the creep rate was only 0.0054 per cent per 1000 hr. at 26,500 psi., a stress far above the yield strength of the metal at this temperature.

The creep resistance of the drawn material is exceptionally good at 500 F. Although its creep rate is greater than for the annealed material for stresses below about 10,000 psi., it is much superior at higher stresses. A stress of 40,800 psi. produced a creep rate of only 0.0035 per cent per 1000 hr. after 4800 hr.

There was no significant change in the structure or tensile properties of the alloy either as annealed or as drawn in testing at 300, 400, and 500 F. It was unique among the alloys investigated, in that it did not recrystallize at all at 500 F. It is well known that this alloy has a higher recrystallization temperature for ordinary annealing periods than most other copper alloys.

EFFECT OF ALLOY COMPOSITION

It is obvious from the test results that the 70-30 copper-nickel has the best creep resisting characteristics of the alloys tested. Quantitative comparisons of the creep resistance of the other alloys are difficult to make because of the importance of the factors of grain size for annealed tempers and of both grain size and degree of final reduction for cold-worked tempers. Additional data are required. However, it appears that naval brass is inferior to 70-30 brass. The silicon bronze is definitely, and the Admiralty metal and phosphor bronze may be slightly, superior to 70-30 brass. The 85-15 brass is in some respects better than the 70-30 brass. It should be remembered that at 400 and 500 F. the silicon bronze suffers an intercrystalline deterioration at the surface, which is independent of stress.

SUMMARY AND CONCLUSIONS

1. Grain size and degree of cold working are important factors in determining the creep resistance of these copper alloys, and in general are fully as important as chemical composition.

2. Creep resistance of annealed 70-30 brass for feasible stresses increases with grain size at 300, 400, and 500 F. This is partly in agreement with previously published information, but definitely lowers the known temperature at which coarser grained metal becomes superior to fine-grained metal of this composition.

3. The creep resistance of these alloys, finished by a cold working operation, is largely determined by their recrystallization characteristics. If they recrystallize within a short time at elevated temperature, they behave as annealed materials. If recrystallization takes place over a prolonged period, the materials may be very unstable with regard to creep. If no recrystallization occurs, the creep resistance may be high, but on the other hand, intercrystalline parting may embrittle the metal. In the last case recrystallization would probably begin after an exceedingly long time.

4. Very coarse grained metal, either as annealed or as cold worked, may become embrittled and in danger of sudden failure under conditions where it appears to offer high resistance to creep.

5. The structure of naval brass at 300, 400, and 500 F. is characterized by the formation of a third phase, presumably gamma.

6. The wisdom of extending the creep testing period as long as possible is clearly evident.

7. The 70-30 copper-nickel alloy is easily the best of the alloys tested for service at elevated temperatures.

Acknowledgment:

The authors wish to make due acknowledgment to D. K. Crampton for his interest and suggestions concerning the investigation, to M. L. Wood and L. E. Thelin for their suggestions regarding the development of the furnaces, to F. P. Strieter for assistance in conducting part of the investigation, and to other members of the Research Department of the Chase Brass and Copper Co. who helped in any way.

REFERENCES

(1) C. L. Clark and A. E. White, "Properties of Non-Ferrous Alloys at Elevated Temperatures," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 53, p. 183 (1931).

(2) C. L. Clark and A. E. White, "Influence of Recrystallization Temperature and Grain-Size on the Creep Characteristics of Non-Ferrous Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part II, p. 492 (1932).

(3) A. E. White and C. L. Clark, "Influence of Grain-Size on the High-Temperature Characteristics of Ferrous and Non-Ferrous Alloys," *Transactions*, Am. Soc. Metals, Vol. 22, p. 1069 (1934).

(4) H. J. Tapsell and A. E. Johnson, "Creep Tests at 300 C. and 350 C. of Arsenical Copper and Silver-Arsenical Copper," *Journal*, Inst. Metals, Vol. 48, p. 89 (1932).

(5) H. J. Tapsell, A. E. Johnson, and W. J. Clenshaw, "Properties of Materials at High Temperatures. The Strength at High Temperature of Six Steels and Three Non-Ferrous Metals," *Engineering Research Special Report No. 18*, Department of Scientific and Industrial Research (1932).

(6) W. A. Baker, "Creep of Non-Ferrous Metals and Alloys," *Research No. 58*, British Non-Ferrous Metals Research Assn., June, 1937.

(7) A.S.M.E.-A.S.T.M. Compilation of Available High-Temperature Creep Characteristics of Metals and Alloys (1938).

(8) P. G. McVetty, "The Interpretation of Creep Tests," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 105 (1934).

(9) H. L. Burghoff and C. H. Mathewson, "Time and Temperature Effects in the Deformation of Brass Crystals," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 143, p. 45 (1941).

(10) S. E. Maddigan and A. I. Blank, "Recovery and Recrystallization in Long-Time Annealing of 70-30 Brass," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 137, p. 170 (1940).

(11) O. Bauer and M. Hansen, "The Influence of Third Metals on the Constitution of Brasses. III. The Influence of Tin," *Zeitschrift für Metallkunde*, Vol. 22, p. 405 (1930).

DISCUSSION

Mr. H. B. Oatley[1] (*presented in written form*).—As mentioned by the authors, there has been a comparatively small amount of creep test data for non-ferrous materials and a great deal of what has been made public is not always easily comparable due to a lack of uniformity in procedure, equipment, and methods in the various research laboratories both here and abroad. The progress towards standardization in such procedures, as evidenced by the specifications of the A.S.T.M., should make data that are now becoming available of even greater value because of closer conformity to standard practices.

The increasing use at moderately elevated temperatures of non-ferrous materials has been of increasing and widespread growth in recent years. The need, therefore, of reliable data disclosing creep stress values has become more necessary. The determination in construction codes, of maximum allowable working stresses in order that reasonable safety to pressure vessels making use of non-ferrous materials may be more rational, has been urgently needed. It is hoped that more data of a value comparable to the value of the data contained in the paper under discussion will be forthcoming in the not far distant future.

Mr. J. J. Kanter.[2]—I want to express appreciation for the very useful data which the authors of this paper have made available. It represents an impressive amount of work and indeed is much needed.

There has been some question as to the high temperature usefulness of silicon bronze. The micrograph which the authors show revealing the surface oxidation effect is quite pertinent to interpreting difficulties arising through the use of this alloy.

In interpreting data given for the 70-30 copper-nickel alloy at temperatures of 300, 400 and 500 F., the authors conclude that this alloy has outstanding high-temperature strength. However, there have been serious proposals to standardize working stresses for this alloy up to 750 F. Since the authors have stated the conclusion in their paper that the 70–30 copper-nickel alloy is easily the best of the alloys tested at elevated temperatures, more data bearing on long-time rupture ductility for this material above 500 F. would be desirable. It would be pertinent to have an expression from the authors as to the importance of long-time rupture ductility for copper-base alloys in evaluating their high temperature usefulness under stress.

Mr. H. L. Burghoff.[3]—Mr. Kanter has raised the question of use of 70–30 copper nickel at temperatures up to 750 F. While the results of our tests upon this material show that it has high resistance to creep at 500 F., they should not be extrapolated to higher temperatures. Use at such higher temperatures should be based upon experience on actual test. There are some published creep data[4], based upon tests of 1000-hr. duration, which indicate that the alloy may have useful creep resistance at 750 F.

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[1] Vice-President, The Superheater Co., New York, N.Y.

[2] Materials Research Engineer, Crane Co., Chicago, Ill.

[3] Research Metallurgist, Chase Brass and Copper Co Waterbury, Conn.

Data on long-time rupture ductility would be valuable additions to the creep data on this and any other material, for they would give information on the possibility of sudden and unexpected failure in the case of a material with low long-time rupture ductility or of gradual and predictable failure in the case of a material with high long-time rupture ductility.

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[4] A.S.M.E.—A.S.T.M. Compilation of Available High-Temperature Creep Characteristics of Metals and Alloys (1938).

FATIGUE TESTING OF ZINC-BASE ALLOY DIE CASTINGS

By E. H. Kelton[1]

Synopsis

Fatigue tests were conducted on die-cast specimens of several zinc-base die-casting alloys to satisfy the occasional demands for these values.

The specimens were subjected to three aging conditions before testing: 6 months at room temperature; 6 hr. in 95 C. dry air; and 5 days in 95 C. steam.

The testing was done at 25±1 C. at a speed of 1600 cycles per minute in a flexure-type machine. The majority of specimens were ¼ in. thick but a few were ⅛ in. thick. The results were checked superficially by rotating cantilever-beam tests. The endurance limit was arbitrarily chosen as the greatest stress not causing failure at 100,000,000 cycles. Each point on the *S-N* curve was the first failure in a 20-specimen test at the same stress.

The flexure fatigue limits determined are compared with tensile values:

Treatment	Endurance Limit, psi.		
	Zamak-2	Zamak-3	Zamak-5
Aged 6 months at room temperature	8 500	6 875	8 175
Annealed 6 hr. in dry air at 95 C.	8 000	6 500	7 675
Exposed 5 days in 95 C. steam tank	6 200	5 950	5 700[a]
	Tensile Strength, psi.		
As die cast	52 100	41 000	47 600

[a] Extrapolated value.

The few tests on ⅛-in. specimens failed to indicate a significant difference due to specimen size. The rotating beam tests indicated only that the flexure tests were on the conservative side.

Since the endurance limits were established on the basis of the first failure in 20-specimen tests, it is believed that a factor of safety of 1.20 to 1.30 should be adequate for engineering calculations.

The extended use of zinc-base die castings has resulted in their occasional application as members subjected to repeated stresses. To aid in designing such members and also to provide a basis of comparison for alloy development work, it was decided to determine the fatigue properties of die-cast Zamak-2, -3 and -5[2] after various treatments.

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[1] Research Division, The New Jersey Zinc Co. (of Pa.), Palmerton, Pa.

[2] The compositions of Zamak-2, -3 and -5 compare with those of A.S.T.M. alloys Nos. XXI, XXIII, and XXV, respectively (A.S.T.M. Tentative Specifications for Zinc-Base Alloy Die Castings (B 86 – 41 T), 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 544). The compositions were as follows:

	A.S.T.M. Alloy No. XXI (Zamak-2)	A.S.T.M. Alloy No. XXIII (Zamak-3)	A.S.T.M. Alloy No. XXV (Zamak-5)
Aluminum, per cent	3.5 to 4.5	3.5 to 4.3	3.5 to 4.3
Copper, per cent	2.5 to 3.5	0.10 max.	0.75 to 1.25
Magnesium, per cent.	0.02 to 0.10	0.03 to 0.08	0.03 to 0.08
Iron, per cent, max.	0.100	0.100	0.100
Lead, per cent, max.	0.007	0.007	0.007
Tin, per cent, max.	0.005	0.005	0.005
Cadmium, per cent, max.	0.005	0.005	0.005
Zinc, per cent	remainder	remainder	remainder

A number of different aging conditions were used at first, but because of the limitations of the testing equipment this number was reduced to the three which seemed most important, namely, (1) a room temperature aging of six months, (2) an accelerated aging at 95 C., and (3) an accelerated corrosion treatment in a steam tank.

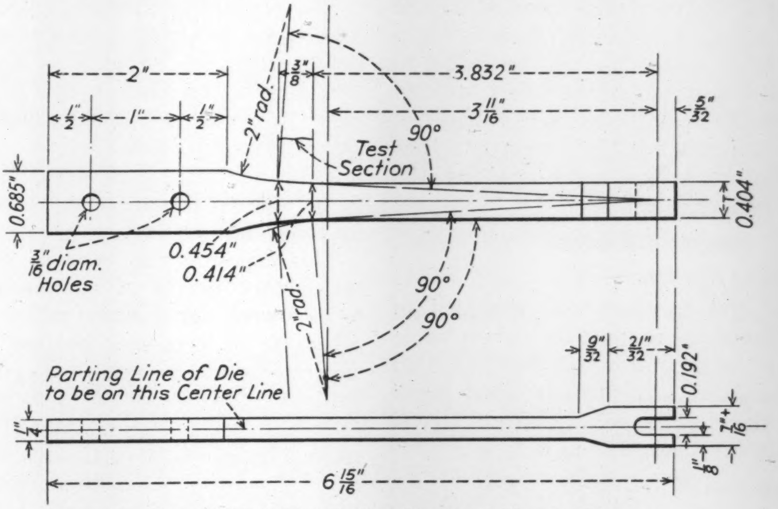
2"
$\frac{1}{2}$"
1"
$\frac{1}{2}$"
2" rad.
$\frac{3}{8}$"
3.832"
$3\frac{11}{16}$"
$\frac{5}{32}$"
90°
Test Section
0.685"
0.404"
T
$\frac{3}{16}$" diam. Holes
0.454"
0.414"
2" rad.
90°
90°
$\frac{9}{32}$"
$\frac{21}{32}$"
0.192"
Parting Line of Die to be on this Center Line
$\frac{1}{4}$"
$\frac{7}{16}$"+
$\frac{1}{8}$"
$6\frac{15}{16}$"

FIG. 1.—$\frac{1}{4}$-in. Die-Cast Fatigue Specimen, Flexure Type.
The 2-in. radius fillets must join the tapered portion without an abrupt break.

Flexure fatigue tests were run at a temperature of 25 ± 1 C. on a machine designed by the Bell Telephone Laboratories, using specimens of $\frac{1}{4}$-in. thickness. A few tests were also run using specimens of $\frac{1}{8}$-in. thickness.

For each alloy and each treatment, tests were run at different stresses until enough points had been obtained to draw the *S-N* curve to at least 100,000,000 cycles. The endurance limit was defined arbitrarily as the greatest stress not causing failure in 100,000,000 cycles. In some cases it was not possible to draw a smooth curve because of erratic results caused by defects such as bent specimens, corrosion (in some steam exposure specimens), and other stress raisers such as notches, scratches, and porosity. In these cases it was still possible to establish a tentative endurance limit.

For comparison with these results, a few rotating-beam fatigue tests were run at a temperature of 25 ± 1 C. on a cantilever-type machine designed by the Bell Telephone Laboratories. The specimens were nominally of $\frac{1}{4}$-in. diameter.

Conclusions:

The flexure fatigue endurance limits fo the three alloys for three types of treatment were determined to be as follows. Tensile values are shown for comparison.

Treatment	Endurance Limit, psi.		
	Zamak-2	Zamak-3	Zamak-5
Aged 6 months at room temperature	8 500	6 875	8 175
Annealed 6 hr. in dry air at 95 C.	8 000	6 500	7 675
Exposed 5 days in 95 C. steam tank	6 200	5 950	5 700[a]
	Tensile Strength, psi.		
As die cast	52 100	41 000	47 600

[a] Extrapolated value.

Since 20 specimens were tested under each set of conditions (alloy, treatment, stress), and since the above endurance limits were derived from the *first* failure in each test group (that is, the poorest specimen of the 20 tested), it was thought that the above figures were conservative and that a safety factor of 1.20 to 1.30 should be adequate for engineering calculations.

The above data were checked with a small number of flexure fatigue tests on $\frac{1}{8}$-in. thick specimens. These failed to indicate any significant effect due to differences in specimen size. The few rotating-beam tests indicated only that the flexure fatigue data were on the conservative side.

FLEXURE FATIGUE TEST

Design of Specimen:

The test specimen was designed so that the stress reached its highest value in the test section, which was a portion of a uniform strength cantilever beam and therefore had a uniform stress. Details are shown in Fig. 1.

Preparation of Specimens:

Casting.—The specimens were die cast in a commercial plunger-type machine operated at a pressure of 1500 psi.

A considerable amount of die development work was done before sound specimens of uniform strength could be cast. This involved principally changes in gates, overflow wells, and water cooling. It was also necessary to alter the shape of the cavity in order to insure that a majority of fatigue breaks would occur in the test section.

Metal and die temperatures were controlled for optimum casting conditions as follow:

	Zamak-2	Zamak-3	Zamak-5
Metal temperature ..	410±5 C.	410±5 C.	410±5 C.
Die temperature	245±5 C.	185±5 C.	185±5 C.

Aging.—If the alloys had been tested immediately after casting, aging changes affecting the dimensions and physical properties of the casting would have caused unstable test conditions. Therefore the specimens were aged, before testing, in one of three ways: (1) at room temperature (17 to 33 C.), for 6 months; (2) in dry air at 95 C., for 6 hr.; or (3) in an atmosphere saturated with moisture at 95 C. and at atmospheric pressure, for 5 days.

The 6-hr. annealing in dry air at 95 C. was chosen as one of the exposures for two reasons:

1. It is a standard stabilizing treatment to overcome the shrinkage which normally requires 6 to 8 weeks.
2. In the testing work it was desirable to be able to prepare many specimens for immediate testing.

This exposure was believed to be a satisfactory means of eliminating the 6- to 8-week aging period which would otherwise be necessary because of the effect of shrinkage during the endurance test.

In the original program of fatigue testing, exposure to 95 C. steam for 10 days (the usual accelerated aging and corrosion test) was included. However, the test results on specimens exposed for even 5 days in steam were somewhat erratic, probably due to the notch effect caused by surface corrosion. It was concluded therefore that Zamak die castings subject to fatigue load would not be suitable if the exposure approached the 10-day steam test in severity. It was desirable, however, to include a moderately severe exposure, and the 5-day steam test seemed to fill this requirement. It is believed that this accelerated exposure test corresponds to many years of severe service.

The steam exposure caused both accelerated aging and corrosion. It was a special case of exposure to steam and hot water, the latter resulting from

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continuous condensation in the exposure chamber.

In all cases the die-casting fins were removed carefully; otherwise, the critical sections of the specimens were tested n the original die-cast condition as ltered by the exposure treatment.

Plating of Yokes.—After aging treatments, the yokes of the flexure fatigue pecimens were given a chromium plating (0.001 to 0.002 in.—sufficient to insure good fit on pin) to prevent excessive wearing by the pins on the oscillating bars, which applied the stress to the specimens.

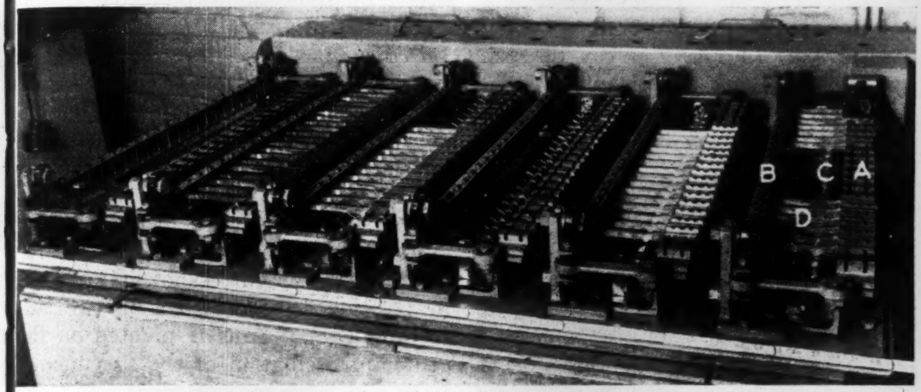

FIG. 2.—Flexure Fatigue Machine, Front View.

A. Clamping bar.
B. Oscillating bar.
C. Counter bar.
D. Test specimens.

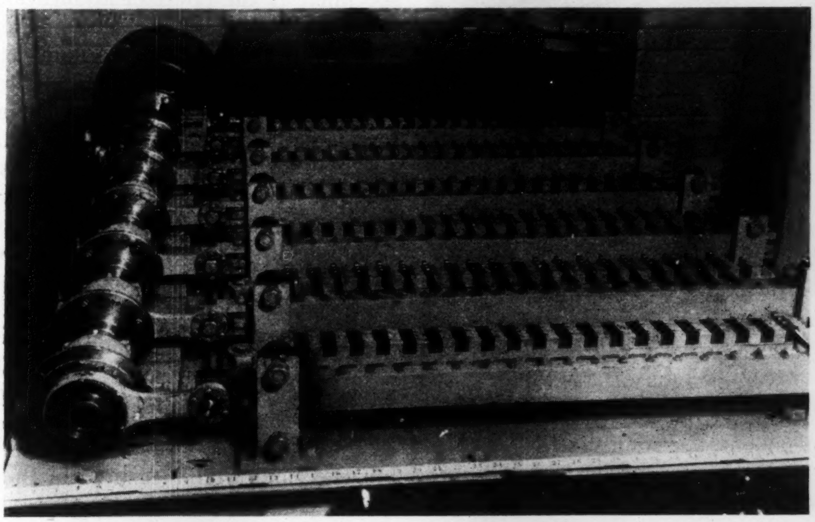

FIG. 3.—Flexure Fatigue Machine, Side View.

Description of Testing Equipment:

Machine.—Testing was done in a flexure fatigue machine designed and built by the Bell Telephone Laboratories for testing lead alloys. It was modified

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by The New Jersey Zinc Co. (of Pa.) for testing the more rigid zinc-base die-casting alloys. The testing was done at a temperature of 25 ± 1 C. and at a speed of 1600 cycles per minute.

Figures 2, 3, and 4 show the flexure fatigue machine developed to test zinc-base alloys. In Fig. 2 it can be seen that this machine contained six independent units, each consisting of a clamping bar *A*, an oscillating bar *B*, and a counter bar *C*. Each unit tested 20 specimens, *D*, simultaneously. The grip ends were securely fastened into the stationary clamping bars by means of wedges and shims. The opposite ends were flexed by the oscillating bars. In turn, the ends of the oscillating bars were crank-connected to the ends of the clamping bars and moved in the arc of a circle. The force was applied to the specimens by pins in the oscillating bars which engaged the yokes on the ends of the specimens.

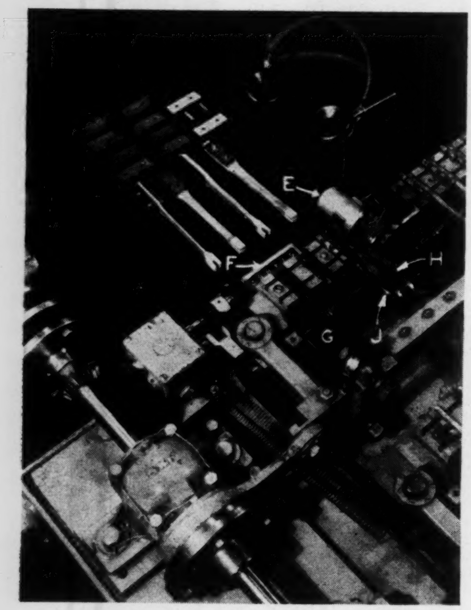

FIG. 4.—Deflection Measurement Device.

E. Dry cell.
F. Master counter.
G. Counter bar.
H. Calipers.
J. Specimen.

Figure 3 shows the drive mechanism. Reciprocating motion was applied to the oscillating bars by cranks which fitted over adjustable cams connected to the continuous drive shaft.

Deflection Measurement Device.—The essential observations in the flexure fatigue test were the amplitude of the deflection given to the specimens and the number of flexures required to cause failure.

The deflections were measured with an instrument especially designed for this purpose. Figure 4 shows it mounted in position. It consisted of two adjustable caliper arms *H* pivoted on the center of oscillation of the specimen *J* upon which the measurement was to be made. The arms were fitted with two sets of pins, one set to contact the specimen at the point of application of the force and the other set to measure the caliper opening. The contact pins were part of an electrical circuit, containing a dry cell *E* and head phones, for detecting the instant of contact. The measuring pins were conveniently positioned about two-thirds of the distance from the pivot to the contact pins. A micrometer measurement on the outside of these pins was used to compute the deflection at the point of application of the force. The caliper arms, switch, dry cell, etc., were mounted upon a support plate fastened into the clamping bar of the machine in the same manner as the specimens.

Deflections were measured in the following manner:

1. A test specimen was removed from the bank and a standard specimen was inserted so that the measurement was made with the usual number of specimens being flexed. The standard specimen inserted was a die-cast fatigue specimen of the same thickness as the

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test specimens but not one that was tested, since the yokes of tested specimens sometimes become distorted due to adjustment for wear.

2. The measuring instrument was fastened in the clamping bar with the standard specimen.

3. A zero reading was taken. For this reading, the specimen was stationary and the cam was on its dead-center position. The reading was the distance across the outside of the measuring pins when the caliper arms had been set to barely contact the specimen.

4. A dynamic reading was taken. It was the distance across the same measuring pins when the caliper arms had been set to barely contact the moving specimen.

5. The difference between the readings of (4) and (3), multiplied by the ratio of the distances of the contact and measuring pins from the caliper arm pivot, gave the actual deflection of the specimens at the point of application of the force.

The distance so determined was the total movement of the specimen at the point of application of the force. The deflections, and hence the stresses, were the same in either direction from the unstressed position.

The exact ratio of the distances of the contact and measuring pins from the caliper arm pivot was determined by measurements on ground parallel strips of known thickness. Calibrations were made from time to time to check on the effect of wear on the contact pins. When it became great enough to cause a significant difference in the above ratio in any position, the pins were renewed.

Counting Device.—The number of cycles to failure was recorded by individual Veeder-Root counters mounted on the counter bars (Figs. 2 *C* and 4 *G*). The counter bars oscillated once for every 1000 revolutions of the drive shaft and caused the individual counter arms to contact the bottoms of the unbroken specimens. After a specimen broke, its counter arm no longer made contact and, hence, did not record. The machine was also equipped with a master counter (Fig. 4 *F*) which gave the approximate cycles-to-failure if the individual counters failed to function properly.

Testing Procedure:

Twenty specimens were used as the standard flexure test at any given stress, this being the number that could be accommodated in one bank. The reason for the large number of specimens was that variations in the physical characteristics (porosity, smoothness, straightness, etc.) of the die-cast specimens caused failures to occur over a wide range of cycles.

Only the first failure of each 20-specimen test was used as a point in the determination of the *S-N* curve. Very often a number of failures at a given stress were recorded to give an idea of the range of cycles over which the failures occurred.

Determination of Relationship Between Deflection and Stress:

In order to utilize the data obtained from flexure fatigue tests, the relationship between the deflection of the specimen and the stress in the test section had to be determined.

To accomplish this, a die-cast flexure fatigue specimen (see Fig. 1) was clamped at the grip end to a rigid support and loaded as a cantilever beam. The instantaneous deflection of the yoke end of the specimen was measured by means of an Ames dial for each of a number of loads.

For each load the stress in the outer fibers of the test section was computed by means of the formula:

$$s = P \cdot l \cdot c / I$$

where

s = the stress in the outer fibers in the test section in pounds per square inch,

P = the applied load in pounds,

l = the distance from the point of load application to any given point[3] in the test section in inches,

c = the distance from the neutral axis to the outer fibers in inches, and

I = the moment of inertia at any given point[3] in the test section.

The stress was then plotted against the deflection. Within experimental error this curve was nearly a straight line for the range of deflections used in flexure fatigue testing.

To determine whether the stress-deflection relationship was essentially the same for both static and dynamic conditions of loading, a load-deflection test was run on a rotating-beam machine at 835 rpm. using a die-cast specimen of *uniform* circular section. The details of this apparatus are shown in Fig. 5.

Of course, deflections of the dissimilar flexure and rotating-beam specimens could not be compared directly, so that it was necessary to determine some function of stress and deflection, such as the elastic modulus, that could be used for comparison.

Since die-cast zinc alloys do not exhibit elastic behavior, the modulus, obtained by means of formulas which assume elasticity, has been designated as a pseudo-modulus. The pseudo-modulus decreases with increasing stress and increasing time of stress application; therefore, in both static and dynamic tests the determination was made for each of several loads and as soon after load application as possible.

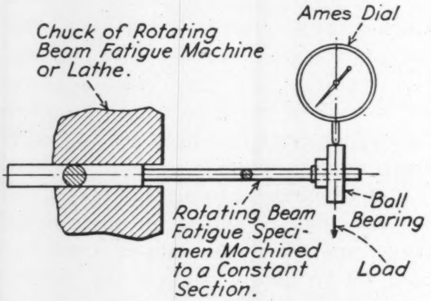

Chuck of Rotating Beam Fatigue Machine or Lathe.
Ames Dial
Ball Bearing
Rotating Beam Fatigue Specimen Machined to a Constant Section.
Load

FIG. 5.—Sketch of Equipment Used to Determine Instantaneous Deformation by the Rotating Beam Method.

The pseudo-modulus of elasticity was calculated for each load by using the formula

$$E_p = P \cdot l^3 / 3\, I_a \cdot d$$

where

E_p = the pseudo-modulus of elasticity in pounds per square inch,

P = the applied load in pounds,

l = the length of beam acting as a cantilever in inches,

d = the deflection at the point of load application in inches, and

I_a = the average moment of inertia[4] of the beam.

The pseudo-modulus was then plotted against stress.

---

[3] Since the test section is a portion of a uniform strength cantilever beam, the stress and the term $l:I$ are constant in this region.

[4] A steel flexure fatigue specimen (whose modulus of elasticity was known) was loaded as a cantilever and the deflection measured. Then the average moment of inertia for the beam could be calculated,

$$I_a = P \cdot l^3 / 3\, E_{steel} \cdot d$$

which would be valid for a Zamak specimen of the same shape.

A comparison of the pseudo-modulus *versus* stress curves was made and, as shown in Fig. 6, the differences were well within the experimental errors of the determinations.

It was assumed, therefore, that the stress-deflection relationship, as determined by static methods, was applicable to the dynamic conditions of the flexure fatigue test.

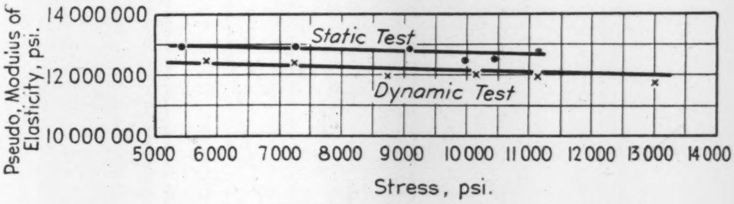


FIG. 6.—Pseudo-Modulus of Elasticity *versus* Stress for Zamak-3.
Aged 6 months at room temperature obtained by static and dynamic load-deflection measurements.

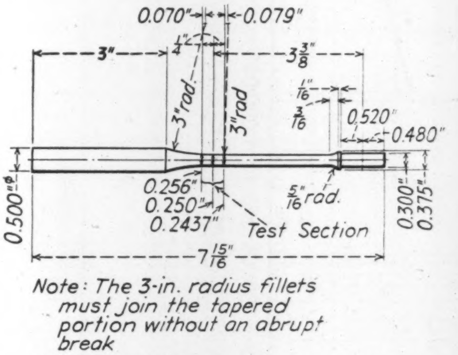

0.070"
0.079"
3"
1/4"
3 3/8"
3" rad.
3" rad
1/16"
3/16
0.520"
0.480"
0.500"
0.256"
0.250"
0.2437"
5/16" rad.
Test Section
0.300"
0.375"
7 15/16"
Note: The 3-in. radius fillets must join the tapered portion without an abrupt break

FIG. 7.—$\frac{1}{4}$-in. Die-Cast Fatigue Specimen, Rotating Cantilever Type.
The 3-in. radius fillets must join the tapered portion without an abrupt break.

ROTATING BEAM FATIGUE TEST

Design of Specimen:

The test specimen was designed so that the stress reached its highest value in the test section, which was a portion of a uniform strength cantilever beam and therefore had a uniform stress. Details are shown in Fig. 7.

Preparation of Specimens:

The specimens were prepared the same as was done in the Flexure Fatigue Test.

Description of Testing Equipment:

Machine.—The machine was designed by the Bell Telephone Laboratories and built by The New Jersey Zinc Co. As shown in Figs. 5 and 8, it consisted of eight rotating chucks *A* which were geared to the drive shaft. After the specimen was mounted in the chuck the outer end was loaded by suspending a weight upon the outer race of a ball bearing containing a bushing which fitted over the specimen end.

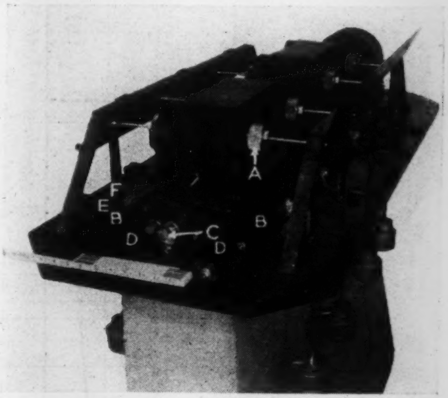
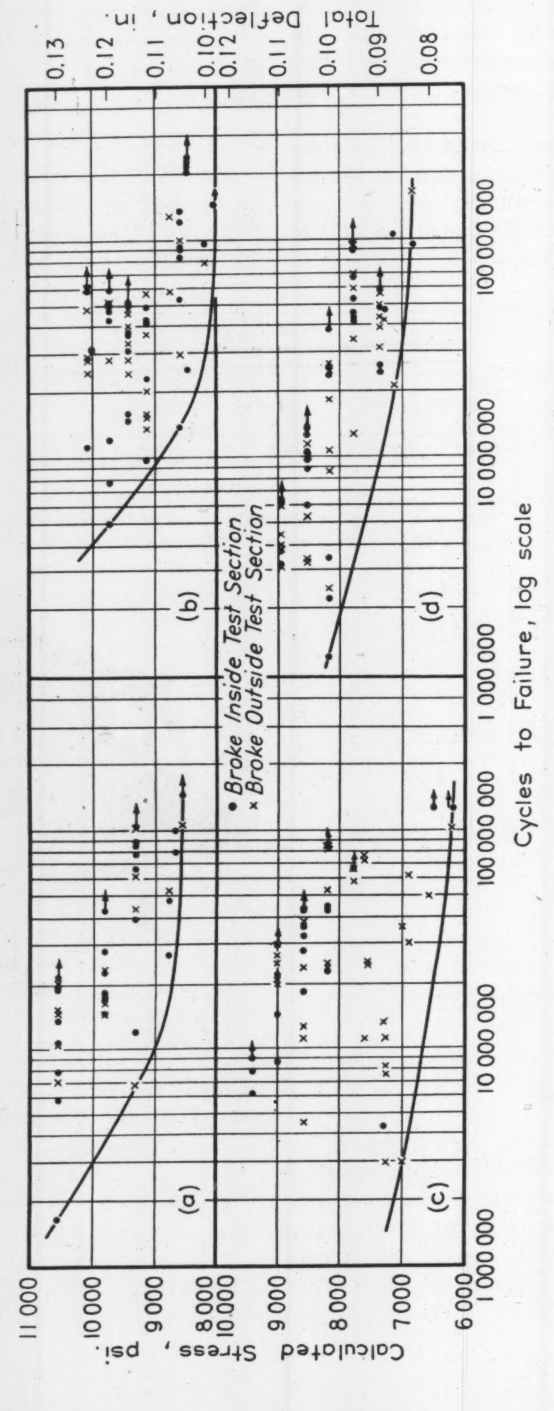


FIG. 8.—Rotating Beam Fatigue Machine.
A. Rotating chucks.
B. Counter Bar.
C. Crank.
D. Connecting rod.
E. Stop.
F. Disk.

Counting Device.—The number of


Calculated Stress, psi.
11 000
10 000
9 000
8 000
10 000
9 000
8 000
7 000
6 000
Total Deflection, in.
0.13
0.12
0.11
0.10
0.12
0.11
0.10
0.09
0.08
(a)
(b)
(c)
(d)
● Broke Inside Test Section
× Broke Outside Test Section
1 000 000
10 000 000
100 000 000
1 000 000
10 000 000
100 000 000
Cycles to Failure, log scale

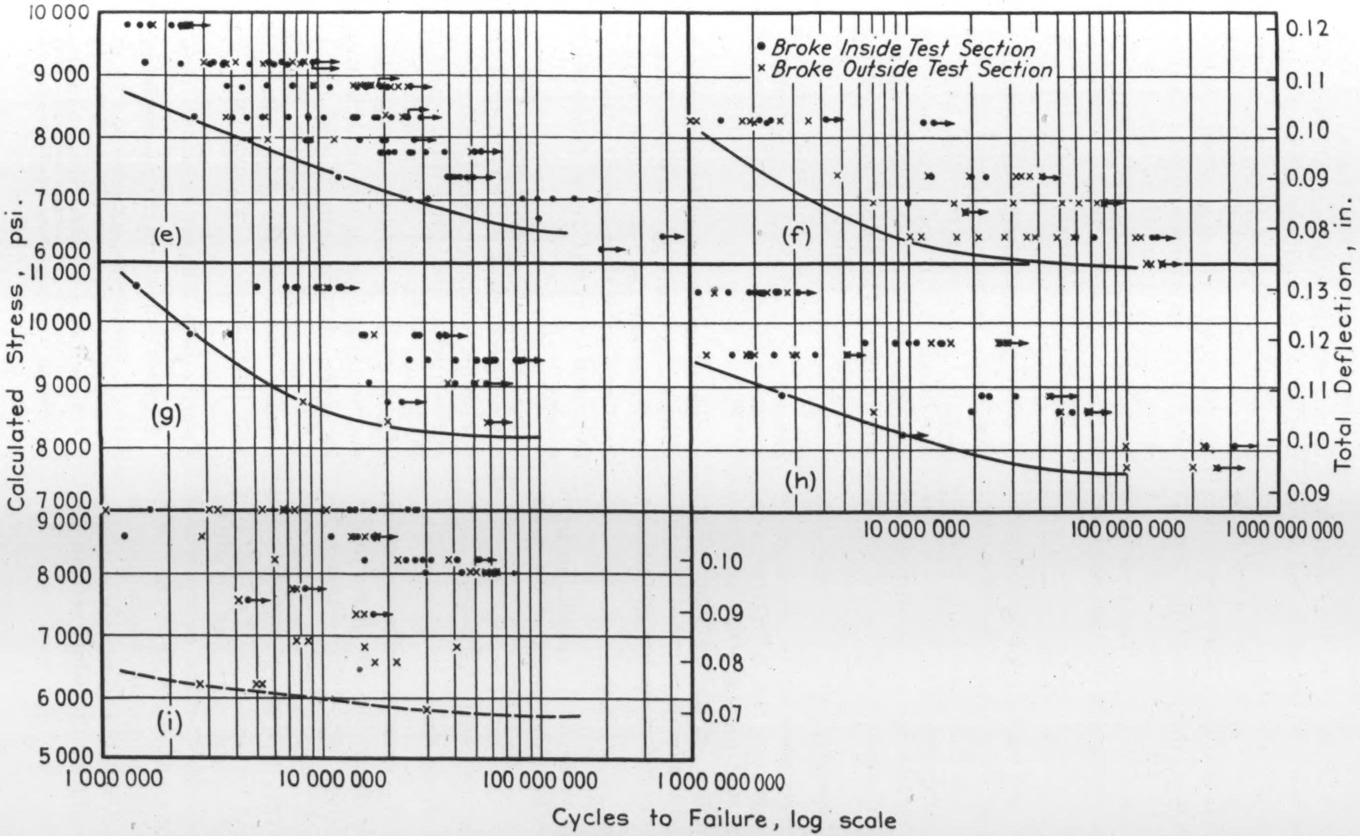


FIG. 9.—Flexure Fatigue Tests, $\frac{1}{4}$-in. Specimens.

(*a*) Zamak-2 aged 6 months at room temperature.
(*b*) Zamak-2 annealed 6 hr. at 95 C. in dry air.
(*c*) Zamak-2 exposed to steam at 95 C. 5 days.

(*d*) Zamak-3 aged 6 months at room temperature.
(*e*) Zamak-3 annealed 6 hr. at 95 C. in dry air.
(*f*) Zamak-3 exposed to steam at 95 C. for 5 days.

(*g*) Zamak-5 aged 6 months at room temperature.
(*h*) Zamak-5 annealed 6 hr. at 95 C. in dry air.
(*i*) Zamak-5 exposed to steam at 95 C. for 5 days.

cycles-to-failure was recorded by individual Veeder-Root counters mounted on counter bars *B* that oscillated about the axis of the counters. The counter bars oscillated once for every 1000 specimen revolutions. They were driven by a crank *C* and connecting rods *D* attached to the low-speed shaft of a 400 to 1 speed reducer. The high-speed shaft was connected to the machine-drive shaft.

As long as the specimens remained unbroken, the oscillation of the counter bars caused the counter arms to contact a stop *E* which actuated the counter. When the specimen was broken, a disk *F* on the weight rod depressed the stop so that the counter arm swung freely.

Testing Procedure:

Eight specimens were used as a standard rotating beam fatigue test, this being the number that could be accommodated in the machine. They were tested at a temperature of 25 ± 1 C. at a speed of 835 cycles per minute.

Determination of Stress:

Stresses were readily computable from the direct load by means of the beam formula, $s = M \cdot c/I$.

RESULTS AND DISCUSSION

Flexure Fatigue Tests:

The *S-N* curves for the individual alloys and treatments are shown in Fig. 9. All curves were drawn through the "first-break" points, that is, the points representing the first specimens to fail in 20-specimen tests. Since the principal object of the work was to obtain endurance values for use in engineering calculations and since there is practically no background of information on the fatigue of zinc die-casting alloys, this conservative procedure seemed justified.

The die-cast specimens used for these tests were the strongest and soundest that could be produced after an extensive die development program. Therefore, when these data are used in design calculations for commercial die castings, which may be porous and contain other stress raisers, it would be advisable to use a factor of safety of, perhaps, 1.20 to 1.30.

The curves showed clearly that, for the aging condition tested, Zamak-2 in every case had the highest endurance limit and Zamak-3 the lowest, with Zamak-5 in between the other two. It was interesting to note that the tensile strengths of the alloys also fell in this same order. The tensile values of die-cast specimens follow:

	Tensile Strength, psi.
Zamak-2	52 100
Zamak-3	41 000
Zamak-5	47 600

The aging treatment of 6 months at room temperature gave the best fatigue properties and the 5-day steam exposure treatment gave the poorest.[5] Stabilization for 6 hr. at 95 C. gave intermediate results.

A few flexure fatigue tests, Fig. 10, run on specimens $\frac{1}{8}$ in. thick showed the same order and range of endurance limits as those determined using specimens $\frac{1}{4}$ in. thick, and indicated that this range of specimen size apparently had no effect on the fatigue properties in this case. However, these results should not be taken as conclusive because the endurance limits of the $\frac{1}{8}$-in. specimens were determined by extrapolation.

Rotating-Beam Fatigue Tests:

Figure 11 shows rotating-beam fatigue data on specimens aged 5 days in dry

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[5] For steam exposure specimens of Zamak-5 it has not as yet been possible to determine accurate endurance limits because of corrosion effects caused by the treatment

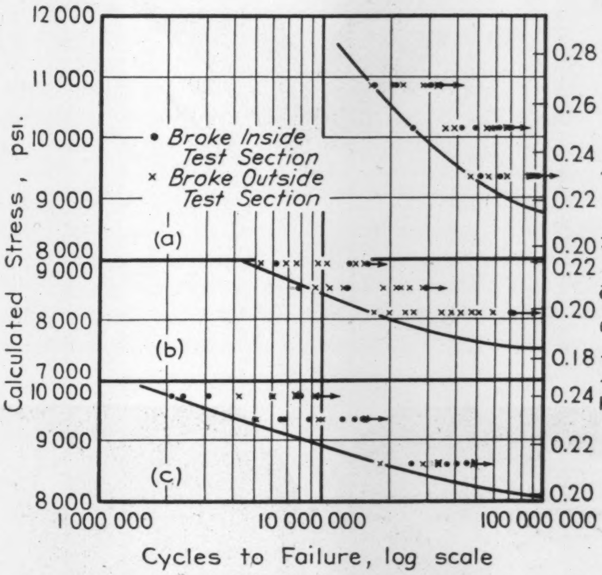


FIG. 10.—Flexure Fatigue Tests, ⅛-in. Specimens Aged 2 yr. at Room Temperature.
(*a*) Zamak-2. (*b*) Zamak-3. (*c*) Zamak-5.

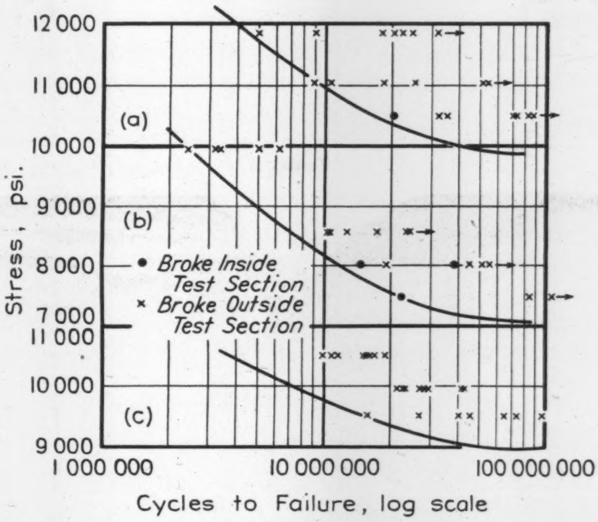
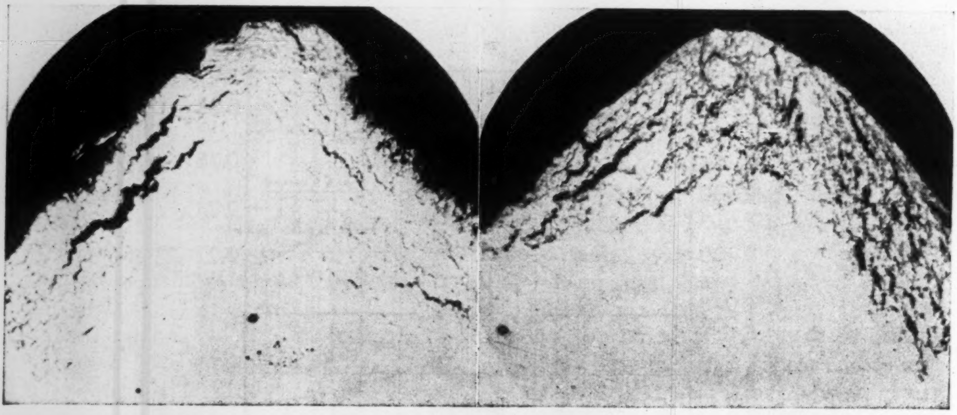
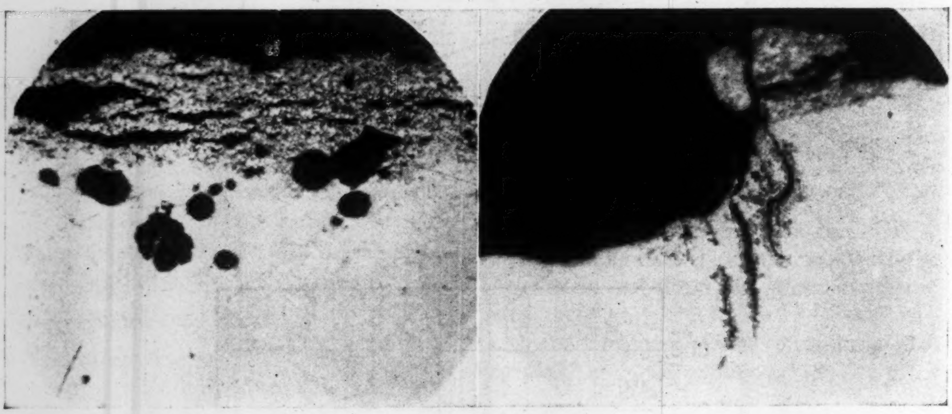


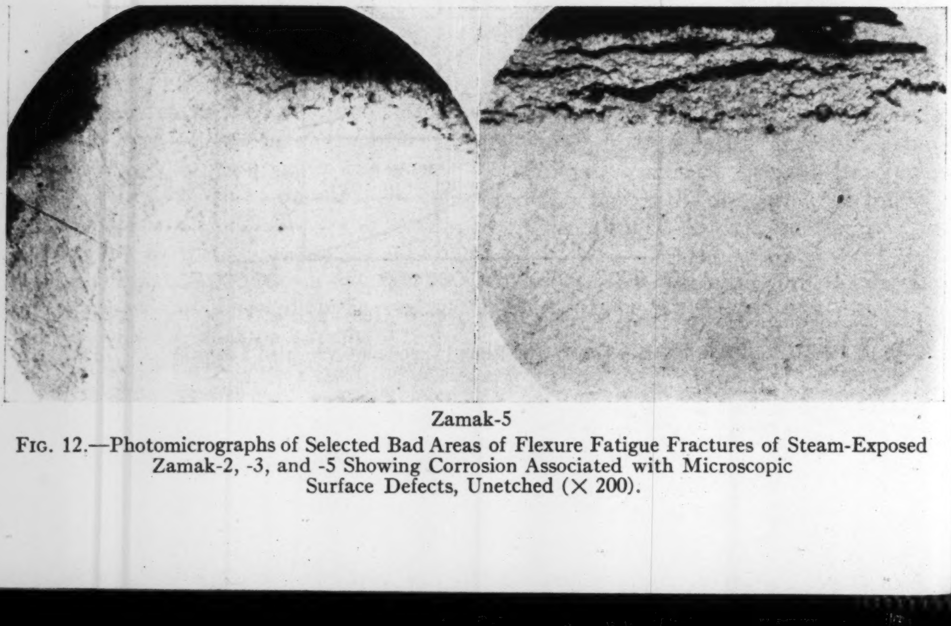
FIG. 11.—Rotating-Beam Fatigue Tests, ¼-in. Round Specimens Annealed 5 days at 95 C. in Dry Air.
(*a*) Zamak-2. (*b*) Zamak-3. (*c*) Zamak-5



Zamak-2



Zamak-3



Zamak-5

FIG. 12.—Photomicrographs of Selected Bad Areas of Flexure Fatigue Fractures of Steam-Exposed Zamak-2, -3, and -5 Showing Corrosion Associated with Microscopic Surface Defects, Unetched (× 200).

air at 95 C. These tests were run as a check on the flexure fatigue data and, as the work was preliminary in nature, some extrapolation was needed to determine the endurance limits. The comparison of these data with flexure fatigue data showed that the order of endurance limits was the same but that the rotating-beam tests gave somewhat higher values. It would not be advisable, in view of the fact that these results were obtained by extrapolation, to use them in design calculations. The more conservative flexure fatigue data should be used.

Causes of Inconsistent Data:

As can be seen by an examination of the *S-N* curves there was a wide spread of data for tests run at all stresses and, in some instances, tests failed after fewer cycles than those run at higher stresses. There were also a large number of failures which occurred outside the test section.

Many of these "fluke" results were presumably caused by specimens being bent when hot, superimposing a stress on that produced by the specimen deflection, and by small notches and scratches on the surface of the specimens, acting as stress concentrators.

In a further effort to determine the causes of the widely different fatigue properties that were exhibited by castings prepared under apparently equivalent conditions, radiographic and surface orientation tests were made on a number of specimens.

All of the specimens that were examined radiographically were found to be reasonably sound, and the small differences in the amount of porosity that did exist could not be correlated with differences in fatigue strengths. Surface orientation tests also failed to show any correlation with fatigue strength.

The flexure fatigue specimens which gave the most erratic results were the steam exposure specimens. Therefore, microscopic examination was made of a number of these erratic specimens at the plane of fracture without etching. Shallow corrosion was found in occasional spots, apparently associated with microscopic surface defects in the original specimens and the fact that in the steam test corrosion is sometimes localized due to condensation. These are shown by the photomicrographs in Fig. 12. The corrosion has enlarged these defects and apparently has magnified their effect as stress raisers.

This condition was found in all of the Zamak alloys and quite probably was the cause of many inconsistent data.

Acknowledgment:

The work described in this paper was carried out over a period of 8 yr. by the Research Division of The New Jersey Zinc Co. (of Pa.). A number of investigators took part in the work. Particular acknowledgment is due to Gerald Edmunds, who took a major part in the design of the apparatus and development of the test procedure; and to L. J. Held, M. D. Brewster, J. F. Wisler, and P. W. Ramsey, who obtained most of the experimental data.

DISCUSSION

MR. G. W. STICKLEY[1] (*presented in written form*).—This paper undoubtedly is a valuable contribution to the information concerning fatigue properties. Although in the past repeated stresses seldom have had to be considered when zinc-base die castings were used, a knowledge of the fatigue strengths of these alloys should be a real aid to the designer. It should be remembered, however, that the actual fatigue strength of a die casting depends considerably upon the actual design, as for example, when grooves and fillets are used. Also, as the author indicates, the fatigue strength is affected by the design of the die and by the casting procedure.

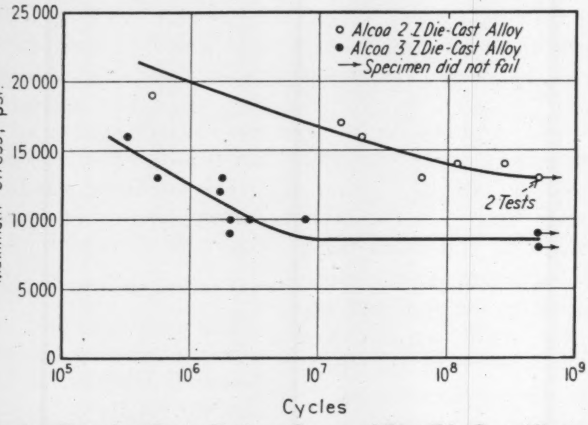


FIG. 1.—Rotating-Beam Fatigue Curves of Zinc Die-Cast Alloys.

The endurance limits obtained in the rotating-beam tests of each alloy are considerably higher than the corresponding values obtained in the flexural tests. This may be attributed to a number of causes. Among them may be the difficulty of casting specimens of the rectangular cross-section in such a manner as to have them free from casting strains at the corners. It is also more difficult to obtain a satisfactory distribution of stress in the type of flexure test described than in a rotating-beam test, because of bent specimens and slight misalignments.

Several years ago the Aluminum Research Laboratories made some tests of Alcoa 2Z and 3Z alloys which are similar to Zamak-2 and -3. In these tests all specimens were radiographed and the sound specimens were selected and tested. The tests were made in R. R. Moore rotating-simple-beam fatigue machines using specimens having a diameter of 0.330 in. in the center of the reduced section. The results of these tests, which are shown in the accompanying Fig. 1, indicate higher endurance limits than those reported by Mr. Kelton. The corresponding tensile strengths were 47,600 and 36,300 psi.

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[1] Aluminum Research Laboratories, Aluminum Company of America, New Kensington, Pa.

MR. E. H. KELTON.[2]—Mr. Stickley's comments are much appreciated, especially since his work indicated higher endurance limits than ours. In our own work perhaps we have been too conservative. On the other hand, in the early determinations of values of this type, realizing that these values may be used in engineering calculations, little harm will be done by interpreting all tests on the safe side.

In examining Mr. Stickley's data, it will be noted that the curves are based on 7 and 10 points, respectively, for Alcoa 2Z and 3Z. It is assumed that each point represents a single specimen. In our investigation, 40 and 58 points of failure are shown, respectively, for similar alloys (aged six months at room temperature) and at least an equal number of specimens was removed from the test before failure since a "first break" for a given deflection had been established. It will also be noted by reference to Fig. 9 that first tests at relatively high deflections sometimes indicated endurance limits somewhat higher than those eventually established by tests at lower deflections. It has not been determined whether this was due to slight variations from one cast to another or variations in the test itself. It was felt, however, that these unexplained variations were not greater than would be encountered in commercial castings and that they should be recognized.

I believe that with an equal number of tests and similar interpretation, the differences between our tests and Mr. Stickley's would be much less.

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[2] Research Division, The New Jersey Zinc Co. (of Pa.), Palmerton, Pa.

HARDNESS CONVERSION RELATIONSHIPS

By Robert H. Heyer[1]

Synopsis

The conversion relationships which are now in general use for materials in the Rockwell "B" range of hardness are not satisfactory for certain classes of materials, such as aluminum and its alloys and the austenitic stainless steels. They apply very satisfactorily to yellow brass.

In order to study the relationships between hardness scales based on pyrammid and ball types of penetrators, the following tests were made on a variety of materials: Vickers 10 kg., 30 kg., and 50 kg., Rockwell "B" and "F", Rockwell Superficial 45-T, 30-T, and 15-T. In addition to the standard procedures, the diameters of all Rockwell impressions were measured with the Vickers microscope in order to establish work-hardening capacities by Meyer's analysis.

Special Rockwell tests were made to compare rates of flow under load in the hardness test.

Three factors which contribute to the uncertainty of present hardness conversion relationships when applied to a variety of materials having hardnesses in the Rockwell "B" range were found to be:

1. Differences in work-hardening capacities,
2. Differences in the contours of the hardness impressions,
3. High rates of flow under load in the hardness test.

Of these factors, the first is in most cases the most important.

This investigation was undertaken to improve the reliability of hardness conversion relationships in the Rockwell "B" range. The work is closely related to the activities of the Subcommittee on Hardness Conversions of the Section on Indentation Hardness of the Society's Committee E-1 on Methods of Testing, and most of these data have been presented informally before this group.

The purpose of this preliminary report is to point out some of the factors which have made hardness conversions in this range somewhat unreliable. Further work is contemplated for the purpose of establishing definite conversion relationships for use with specific materials in the Rockwell "B" range.

Influence of Time of Load Application:

Many materials having hardnesses in the Rockwell "B" range have the characteristic of flowing under load in the hardness test, thus the time factor must be controlled for each type of test before a consistent conversion can be expected. The following experiments were made to show the importance of this factor.

Test specimens of brass, duralumin,

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[1] Metallurgist, The American Rolling Mill Co., Middletown, Ohio.

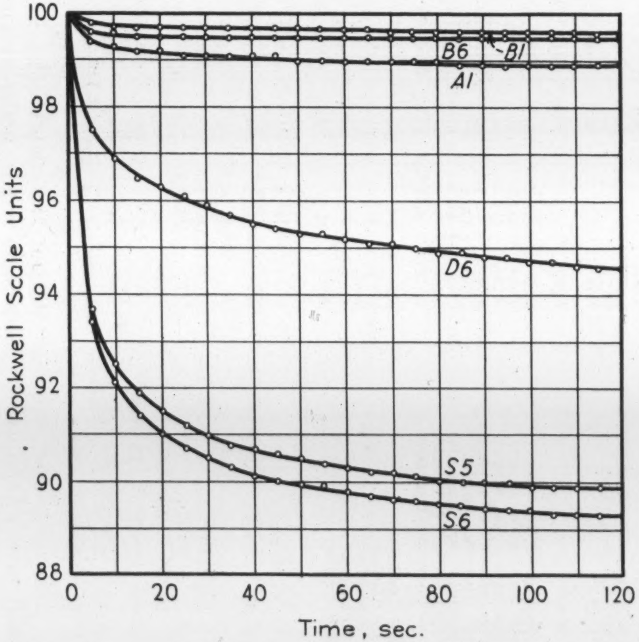

FIG. 1.—Relative Rates of Flow of Six Metals in Terms of Rockwell "B" Scale Units and Time of Load Application.

	B1	*B6*	*A1*	*D6*	*S5*	*S6*
Rockwell "B"	31	7	66	7	29	33
Reading after 120 sec. load application	31	7	65	−2	22	25
Meyer "*n*"	2.35	2.46	1.97	2.05	2.46	2.29

TABLE I.—EFFECT OF TIME OF LOAD APPLICATION IN A ROCKWELL HARDNESS TESTER.

Time, sec.[a]	DIAL READINGS ON THE RED OR "B" SCALE[b]					
	Specimen B1	Specimen B6	Specimen A1	Specimen D6	Specimen S5	Specimen S6
0	102.6	80.8	137.5	75.9	106.7	110.4
5	102.4	80.4	136.9	73.4	100.4	103.8
10	102.3	80.4	136.8	72.8	99.2	102.5
15	102.3	80.3	136.7	72.4	98.6	101.9
20	102.3	80.3	136.7	72.2	98.2	101.4
25	102.3	80.3	136.6	72.0	97.9	101.1
30	102.3	80.3	136.6	71.8	97.7	100.9
35	102.3	80.3	136.6	71.6	97.6	100.7
40	102.3	80.3	136.6	71.4	97.4	100.5
45	102.3	80.3	136.6	71.3	97.3	100.4
50	102.3	80.3	136.5	71.2	97.2	100.3
55	102.3	80.3	136.5	71.2	97.1	100.3
60	102.3	80.3	136.5	71.1	97.0	100.2
65	102.3	80.3	136.5	71.0	96.9	100.1
70	102.2	80.3	136.5	71.0	96.9	100.0
75	102.2	80.3	136.5	70.9	96.8	100.0
80	102.2	80.3	136.5	70.8	96.7	99.9
85	102.2	80.3	136.4	70.8	96.7	99.9
90	102.2	80.3	136.4	70.7	96.7	99.8
95	102.2	80.3	136.4	70.7	96.7	99.8
100	102.2	80.3	136.4	70.6	96.6	99.8
105	102.2	80.3	136.4	70.6	96.6	99.7
110	102.2	80.3	136.4	70.5	96.6	99.7
115	102.2	80.3	136.4	70.5	96.6	99.7
120	102.2	80.3	136.4	70.5	96.6	99.7
Final Reading	30.7	7.1	65.2	−2.1	22.5	25.3
Rockwell "B"[c]	31.2	7.4	66.2	6.6	29.0	33.3

[a] Time in seconds after the weight arm becomes completely free from the control of the dash pot. The "final reading" was taken upon removing the load after 120 sec. application.

[b] Zero on the red scale taken as 100.

[c] Standard Rockwell "B" readings obtained by releasing the load within 1 sec. after the weight arm becomes completely free from the control of the dash pot.

TABLE II.—IDENTIFICATION OF SPECIMENS AND RESULTS OF VICKERS HARDNESS TESTS.

Specimen	Material	Gage	DPH (Vickers Tester)			
			10-kg. Load	30-kg. Load	50-kg. Load	Average 30 and 50-kg. Loads
A1	Aluminum alloy—rolled	0.056	.....	122	123	122.5
A3	Aluminum alloy—450 F. annealed	0.056	.....	93.6	94.6	94.1
A4	Aluminum alloy—400 F. annealed	0.056	.....	114	110.6	112.3
C1	Copper—rolled	0.126	.....	114	114	114.0
C3	Copper—rolled	0.051	.....	120	120.4	120.2
C7	Copper—rolled 350 F. annealed	0.051	.....	107	106.5	106.7
B1	Brass—Wilson Mech. Inst. Co. block	0.25	.....	72.4	73.2	72.8
B2		0.25	.....	79.8	79.7	79.7
B3		0.25	.....	93.4	92.4	92.9
B4		0.25	.....	102.6	103.6	103.1
B5		0.25	.....	121	122	121.5
B6		0.25	.....	59.7	59.3	59.5
S1	Rimming steel—Box Annealed	0.046	.....	82	82.5	82.2
S2	Rimming steel—Box Annealed	0.046	.....	72	72	72.0
S3	Ingot iron	0.077	.....	69	67	68.0
S4	Rimming steel—Box Annealed	0.046	.....	75	74.3	74.6
S5	Rimming steel—Box Annealed	0.046	.....	70.7	72.4	71.5
S6	Ingot iron	0.072	.....	74	72	73.0
M1	Magnesium alloy	0.75	.....	62.8	63.1	63.0
D1	Alcoa 51S—quenched	0.62	.....	87.5	86	86.7
D2	Alcoa 51S—quenched aged	0.62	.....	125.4	123	124.2
D3	Alcoa 51S—quenched 600 F. annealed	0.62	.....	77.4	78	77.7
D4	Alcoa 51S—annealed—rolled	0.088	.....	79.4	79.7	79.5
D5	Alcoa 51S—annealed—rolled	0.128	.....	76.1	75.4	75.7
D6	Alcoa 51S—annealed—rolled	0.211	.....	70.1	69.9	70.1
D7	Alcoa 17SH	0.50	.....	83.6	82.9	83.3
MM1	Monel metal	0.036	.....	119.5	118	118.7
CB1	Cartridge brass—2 Nos. hard	0.203	125.2	126.5	123.2	124.8
CB2	Cartridge brass—4 Nos. hard	0.160	157.9	158.0	159.1	158.5
CB3	Cartridge brass—6 Nos. hard	0.128	173.0	168.0	171.1	169.5
CB4	Cartridge brass—8 Nos. hard	0.099	190.8	186.8	187.8	187.3
CB5	Cartridge brass—10 Nos. hard	0.078	196.6	193.6	194.4	194.0
CB6	Cartridge brass—400 C. annealed	0.128	86.9	85.9	87.1	86.4
CB7	Cartridge brass—450 C. annealed	0.128	78.6	78.7	77.8	78.3
CB8	Cartridge brass—550 C. annealed	0.128	63.1	61.6	61.9	61.7
SS1	Stainless—Austenitic	0.059	.....	216.4	219	217.7
SS2	Type 430 stainless	0.072	.....	155.2	155	155.1
SS4	Type 302 stainless	0.060	.....	163.5	158.2	160.8
SS5	Type 347 stainless	0.083	.....	146.9	148.4	147.6
SS6	Type 316 stainless	0.126	.....	154	155	154.5
SS10	18.5% Cr, 9% Ni steel	0.121	142.1	139.7	139.2	139.4
SS11		0.120	167.8	171.9	172.5	172.2
SS12		0.116	181.0	179.9	181.2	180.5
SS14		0.109	.....	234.8	236.6	235.7
SS30	16% Cr, 10% Ni steel	0.113	142.0	138.2	135.1	136.6
SS31		0.112	171.4	169.4	165.4	167.4
SS33		0.107	235.0	258.2	246.2	252.2
SS60	17% Cr, steel	0.109	147	143.3	143	143.1
SS61		0.106	159	160	157	158.5
SS62		0.102	174	175	178	176.5
SS63		0.098	186	193.5	195	194.2

low-carbon steel, and ingot iron were used. The Rockwell tester was adjusted for 5-sec. movement according to the recommendation in A.S.T.M. Standard Method of Test for Rockwell Hardness of Metallic Materials (E 18 – 36).[2] Five tests were made under recommended testing conditions, that is, removing the load within 1 sec. after the weight arm was released from the control of the dash pot. Five additional tests were run in which the load was applied for 120 sec. after the weight arm became free. During the course of these tests, readings of the position of the pointer were taken every 5 sec. (Three observers were required to time, read, and record the data.) The average results are summarized in Table I and are illustrated in Fig. 1. The specimens are identified in Table II.

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[2] 1939 Book of A.S.T.M. Standards, Part I, p. 743.

For convenience in comparison, all dial readings have been displaced to 100 for the first or zero time reading.

Comparison of specimens B1 and S6 shows that the flow characteristic is not simply a function of hardness. The low movement in the tests on B6 is further evidence that very soft materials may resist flow. Furthermore, the Meyer "n" values which are included in Fig. 1 show that flow is in no way related to work-hardening capacity.

Test Methods:

A variety of ferrous and non-ferrous test specimens were used for the comparative hardness tests. These specimens are identified in Table II. The surfaces tested were either cold rolled, bright annealed, annealed and pickled, or polished to a No. 00 finish.

The Rockwell and Superficial Rockwell testers were checked against standard blocks and the time of load application adjusted as recommended in A.S.T.M. Standard Method E 18 – 36 and Tentative Method of Test for Rockwell Superficial Hardness of Metallic Materials (E 18 – 39 T).[3] Because of the tendency of some of the materials to flow under load, the time of load application was controlled by removing the major load within 1 sec. after the weight arm was released from the control of the dash pot. A $\frac{1}{16}$-in. diameter ball was used in all tests.

The loading mechanism of the Vickers tester was checked by means of a dead weight - lever type calibrating device. The diamond penetrator used has been carefully checked against a penetrator of exact dimensions by means of comparative hardness tests. The time of load application in the Vickers tests was set for 15 sec.

The data are summarized in Tables II, III, and IV. All hardness values are the average of five observations.

Meyer Hardness Analysis:

The diameters of all Rockwell impressions were measured at 40× or 100× on the Vickers tester. These impression diameters were plotted against the corresponding loads on log-log paper to obtain the work-hardening exponent "n" by Meyer's analysis (**1**, **2**, **3**).[4] From the Meyer relationship

$$L = a\,d^n$$

where

L = load in kilograms,
d = impression diameter in millimeters, and
a and n = constants of the test sample.

It is evident that

$$\log L = \log a + n \log d,$$

whereupon n is the "slope" coefficient of a linear equation in log L and log d. The constant n is readily obtained graphically as the slope of the line drawn through L, d points plotted on log-log paper.

The values of the exponent n vary from approximately 2.0 for cold-rolled materials to 2.5 for some materials in the dead-soft condition. It is obvious that

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1939 Book of A.S.T.M. Standards, Part I, p. 1238.

[4] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 721.

TABLE III.—BRINELL HARDNESS CALCULATED FROM MEYER HARDNESS DATA BASED ON MEASURED DIAMETERS OF ROCKWELL "B," "F," 45-T, 30-T, AND 15-T IMPRESSIONS.

Specimen	DPH, Avg., 30 and 50 kg.	Brinell (1/16 in. = 1.588-mm. Ball = D)					Meyer "n"
		d/D = 0.1 (0.1588-mm. impression diameter)	d/D = 0.2 (0.3176-mm. impression diameter)	d/D = 0.378 (0.60 mm. impression diameter)	L/D^2 = 5 (12.5-kg. load)	L/D^2 = 30 (76-kg. load)	
A1	122.5	126.0	122.2	116.0	121.0	108.3	1.97
A3	94.1	97.3	92.4	87.1	90.0	76.9	1.95
A4	112.3	111.0	108.6	102.2	106.8	93.8	1.97
C1	114.0	108.3	107.4	106.0	107.7	98.8	2.02
C3	120.2	121.0	118.6	114.7	116.8	108.3	2.00
C7	106.7	91.8	100.0	105.0	101.7	103.8	2.13
B1	72.8	49.9	63.1	76.1	70.8	80.0	2.35
B2	79.7	52.5	66.2	80.6	73.2	88.7	2.36
B3	92.9	64.0	78.7	92.5	84.1	98.8	2.31
B4	103.1	77.6	88.6	100.7	92.9	103.9	2.24
B5	121.5	103.3	112.4	119.4	114.3	120.7	2.15
B6	59.5	33.8	44.9	58.7	55.5	68.8	2.46
S1	82.2	57.5	69.3	80.3	76.1	85.9	2.29
S2	72.0	48.4	62.4	76.8	70.6	85.9	2.38
S3	68.0	50.4	62.4	72.7	68.5	78.6	2.30
S4	74.6	54.5	66.8	79.2	73.4	85.9	2.33
S5	71.5	42.8	58.7	75.8	70.3	90.1	2.46
S6	73.0	55.5	66.2	76.8	72.6	82.3	2.29
M1	63.0	49.9	59.9	70.0	68.3	73.0	2.29
D1	86.7	75.1	84.3	90.7	88.0	92.2	2.18
D2	124.2	121.0	126.1	128.0	127.5	126.5	2.08
D3	77.7	82.6	79.9	75.7	77.2	67.0	1.97
D4	79.5	67.0	73.7	73.3	73.4	67.0	2.05
D5	75.7	65.9	69.3	70.4	70.3	65.9	2.08
D6	70.1	59.0	64.9	64.8	64.4	59.0	2.05
D7	83.3	87.7	84.9	80.5	84.0	71.9	1.98
MM1	118.7	80.1	100.0	120.4	105.9	130.5	2.35
CB1	124.8	108.3	116.1	122.9	118.2	122.0	2.13
CB2	158.5	138.8	147.4	153.0	146.0	153.0	2.11
CB3	169.5	145.1	156.0	165.3	156.1	166.5	2.13
CB4	187.3	152.1	166.1	177.4	166.0	177.5	2.15
CB5	194.0	171.4	181.0	189.2	181.0	187.5	2.11
CB6	86.4	55.0	69.9	85.3	77.7	93.0	2.38
CB7	78.3	47.9	62.4	77.2	70.7	85.9	2.40
CB8	61.7	34.3	46.8	62.5	59.0	73.7	2.49
SS1	217.7	166.0	198.5	232.0	191.2	234.0	2.30
SS2	155.1	126.0	143.6	162.0	144.0	166.5	2.23
SS4	160.8	116.0	142.4	170.5	144.0	176.0	2.33
SS5	147.6	88.2	116.1	146.8	121.0	157.0	2.43
SS6	154.5	93.2	119.9	151.3	126.0	162.0	2.41
SS10	139.4	77.6	107.5	143.4	114.0	157.0	2.49
SS11	172.2	133.7	154.9	170.5	153.5	176.0	2.22
SS12	180.5	126.0	153.6	181.0	153.5	186.5	2.30
SS14	235.7	138.7	179.9	228.5	175.2	234.0	2.41
SS30	136.6	94.7	121.1	150.0	126.2	157.5	2.38
SS31	167.4	128.5	153.6	181.0	153.5	186.5	2.29
SS33	252.2	184.0	218.5	256.0	208.0	256.0	2.28
SS60	143.1	108.3	124.9	140.0	128.5	150.0	2.23
SS61	158.5	133.7	147.4	157.0	148.5	160.0	2.16
SS62	176.5	165.2	168.5	174.0	170.0	173.5	2.08
SS63	194.2	191.5	191.0	188.0	191.2	186.5	2.02

TABLE IV.—ROCKWELL HARDNESS VALUES.

Specimen	DPH, Avg., 30 and 50 kg.	Rockwell Hardness Number					Meyer "n"
		"B" Scale	"F" Scale	45-T Scale	30-T Scale	15-T Scale	
A1	122.5	65.6	96.9	43.8	64.1	84.2	1.97
A3	94.1	37.7	85.3	25.3	53.6	79.6	1.95
A4	112.3	56.6	92.8	37.3	59.9	82.4	1.97
C1	114.0	60.1	92.8	39.2	60.8	82.8	2.02
C3	120.2	65.0	95.6	43.3	64.1	84.1	2.00
C7	106.7	57.4	91.2	33.1	56.8	80.0	2.13
B1	72.8	29.9	74.3	3.4	36.2	70.3	2.35
B2	79.7	37.0	78.2	9.5	40.3	72.3	2.36
B3	92.9	50.0	85.2	22.2	48.5	76.5	2.31
B4	103.1	57.4	90.1	30.8	54.5	79.3	2.24
B5	121.5	70.2	97.6	43.4	63.3	83.5	2.15
B6	59.5	6.9	62.3	80.3[a]	21.9	63.5	2.46
S1	82.2	40.4	80.3	17.1	45.7	75.7	2.29
S2	72.0	26.4	71.6	0.4	33.8	69.7	2.38
S3	68.0	23.2	69.1	97.7[a]	32.2	68.6	2.30
S4	74.6	37.0	79.2	9.6	40.9	72.8	2.33
S5	71.5	33.3	74.5	1.0	33.6	68.6	2.46
S6	73.0	31.2	75.1	6.6	37.2	70.9	2.29
M1	63.0	21.5	71.8	1.6	35.8	71.4	2.29
D1	86.7	40.6	82.2	17.2	46.4	75.9	2.18
D2	124.2	69.1	97.9	44.6	63.9	84.9	2.08
D3	77.7	14.0	73.4	7.2	42.8	74.8	1.97
D4	79.5	23.4	76.9	10.0	43.8	74.9	2.05
D5	75.7	17.6	74.1	7.5	42.6	74.9	2.08
D6	70.1	6.6	68.3	97.5[a]	36.2	72.1	2.05
D7	83.3	26.6	78.4	14.5	46.1	76.3	1.98
MM1	118.7	67.8	95.5	37.6	58.9	81.6	2.35
CB1	124.8	68.7	97.2	43.1	63.4	83.5	2.13
CB2	158.5	83.5	104.8	56.6	72.3	87.6	2.11
CB3	169.5	87.9	107.2	60.8	75.0	89.0	2.13
CB4	187.3	91.5	109.3	64.1	76.9	90.2	2.15
CB5	194.0	93.5	110.3	65.3	77.9	90.2	2.11
CB6	86.4	43.8	82.4	15.9	45.1	74.8	2.38
CB7	78.3	34.5	77.2	7.3	38.7	71.8	2.40
CB8	61.7	12.0	64.7	83.4[a]	22.7	64.2	2.49
SS1	217.7	95.0	110.5	63.9	76.3	88.8	2.30
SS2	155.1	80.8	103.1	51.9	68.2	85.5	2.23
SS4	160.8	82.4	102.9	50.3	66.7	84.7	2.33
SS5	147.6	78.4	101.2	46.6	64.3	83.7	2.43
SS6	154.5	81.7	102.9	50.4	67.5	84.7	2.41
SS10	139.4	74.5	98.4	41.6	60.6	81.3	2.49
SS11	172.2	86.2	105.6	54.2	70.4	85.1	2.22
SS12	180.5	90.3	107.6	59.2	73.5	86.9	2.30
SS14	235.7	99.9	113.6	68.6	80.0	90.3	2.41
SS30	136.6	73.0	97.5	40.2	59.6	80.8	2.38
SS31	167.4	83.4	103.7	52.0	68.4	85.1	2.29
SS33	252.2	99.8	113.2	69.5	80.0	90.6	2.28
SS60	143.1	75.2	100.1	46.9	65.6	82.8	2.23
SS61	158.5	81.2	103.9	53.3	70.2	86.1	2.16
SS62	176.5	87.3	107.3	58.8	73.5	87.1	2.08
SS63	194.2	92.4	110.0	64.1	77.9	89.2	2.02

[a] Subtract 100 from these readings (they are below zero n the normal 0 to 100 scale).

materials that have been strain hardened by cold rolling ($n = 2.0$) have a relatively low capacity for hardening by additional cold work, whereas dead soft materials ($n = 2.4$ or 2.5) have a rela-

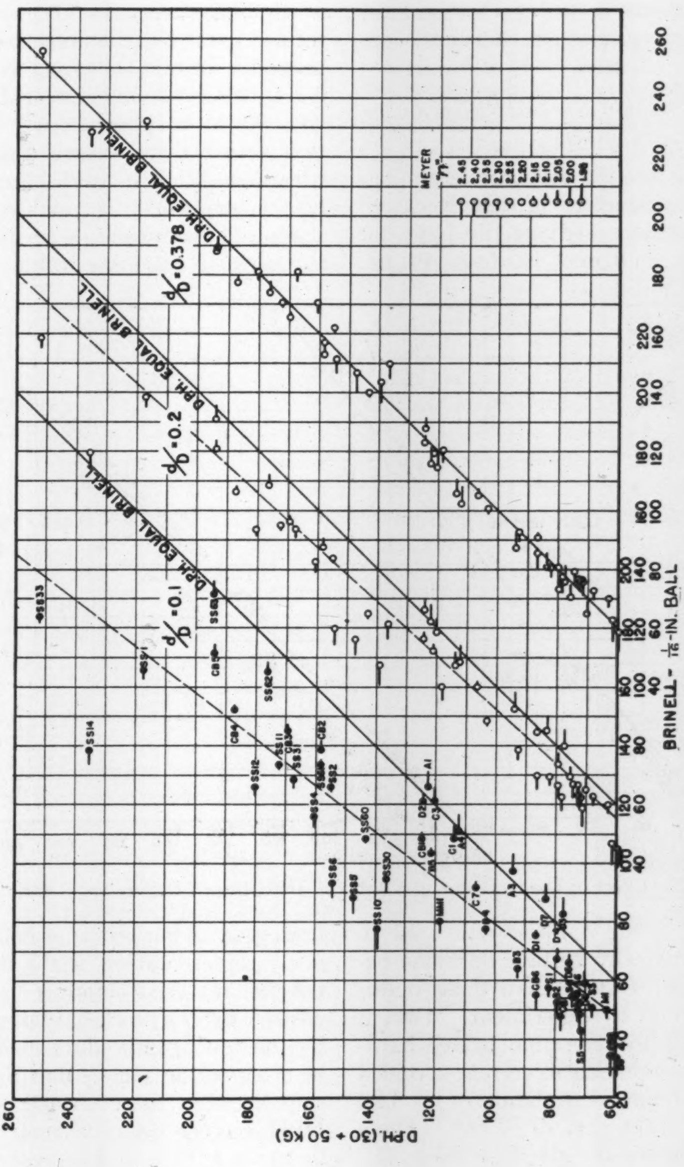

FIG. 2.—Effect of Impression Size on DPH-Brinell Conversion Relationship.

tively high capacity for hardening by cold work.

The exponent *n* will be found most useful in interpreting the hardness conversion relationships.

Diamond Pyramid Hardness-Brinell Conversion Relationships:

In addition to the Meyer *n* determinations, the diameters of the Rockwell impressions were used as the basis for calculation of Brinell hardness values. The log L − log d lines were used to obtain graphically certain relations between L and d other than those determined directly by experiment. Thus it was possible to determine Brinell hardnesses corresponding to certain specified ratios of impression diameter to ball diameter, d/D, or of L/D^2. These hardness values are given in Table III.

In Fig. 2, the Brinell hardnesses corresponding to various ratios of d/D have been plotted against the DPH (diamond pyramid hardness) or Vickers hardness values. The low ratio of $d/D = 0.1$ represents a condition in which the size of the impression is very small with respect to the ball diameter. (The usual range for Brinell testing is from $d/D = 0.2$ to 0.6) From consideration of the geometry of a sphere, it will be evident that very little volume is displaced and consequently little work-hardening will be accomplished in making such a shallow indentation. For this reason the Brinell hardness value calculated from such an impression is believed to be fairly representative of the hardness of the test material before it has been increased by the work-hardening induced by the testing procedure itself. It will be observed, in Fig. 2, that the relationship between these Brinell values and DPH is very poor. Nearly all points lie to the left of a line representing equal values of Brinell and DPH.

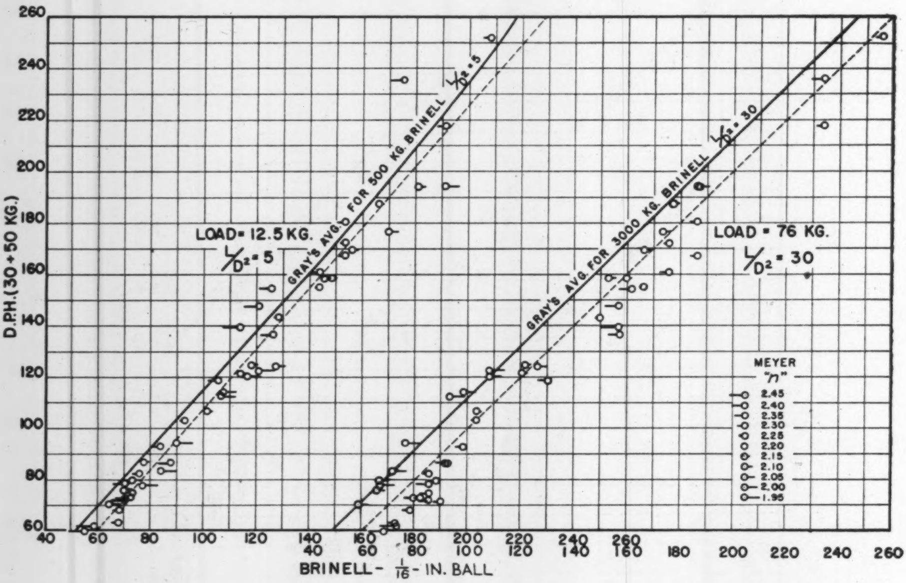

FIG. 3.—Effect of Load/(Diameter)² Ratio on DPH-Brinell Conversion Relationship.

The work-hardening capacities of these materials are represented graphically by the tails on the circled points. According to the legend at the lower right of Fig. 2, a long tail to the right represents a very low work-hardening capacity while a long tail to the left represents a very high work-hardening capacity. Short tails represent intermediate conditions. It is of interest to note that the dotted line makes an almost perfect separation of materials of low work-hardening capacity (to the right) and high work-hardening capacity (to the left).

Specimens A1 and SS12 have the same Brinell hardness, 126, as calculated for an impression diameter of 0.1588 mm. or a ratio of $d/D = 0.1$. The DPH values for these materials are 122.5 for A1 and 180.5 for SS12. The relatively high DPH hardness of SS12 is the result of its high work-hardening capacity ($n = 2.30$). In the Vickers or DPH test, using either 30- or 50-kg. load, the strain hardening induced in the test itself is appreciable, provided the material responds readily to hardening by plastic deformation. In the case of A1 ($n = 1.97$), the response was apparently poor and the DPH value was approximately the same as Brinell hardness at $d/D = 0.1$.

At $d/D = 0.2$, the same trend is evident in the plotted points; however, the dotted line representing intermediate work-hardening rates lies closer to the equality line. In this plot the Brinell hardness of A1 remains practically unchanged, while that of SS12 increases to 154 as a result of the greater work-hardening associated with the larger Brinell impression. (All points in Figs. 2 and 3 can be identified by comparing with points of the same DPH in the $d/D = 0.1$ plot.)

At $d/D = 0.378$ the agreement between DPH and Brinell hardness is quite satisfactory. In general, the materials of high work-hardening capacity are now at the right and those of low n value at the left, suggesting that at a slightly lower ratio of d/D even closer agreement might be found. The Brinell hardness of specimen A1 has decreased to 116, while that of specimen SS12 has increased to 181. The slight decrease in Brinell hardness of A1 for the larger impressions does not indicate true softening but is related to the peculiarities of the Brinell test (see Fig. 3 of reference (3)). The substantial increase in hardness of SS12 is again the result of work-hardening.

From these considerations, it appears that the amount of strain hardening induced in making a Brinell impression with the ratio of $d/D = 0.378$ is closely related to the amount of strain hardening induced in making Vickers impressions with a 136-deg. diamond pyramid penetrator. More specifically, comparable hardness values are obtained under these two test conditions when they are calculated in each case from the usual relationship:

$$\text{Hardness} = \frac{\text{Load}}{\text{Area of the surface of the indentation}}$$

It is probably significant, in this connection, that for a ratio of $d/D = 0.375$ the diameter of the impression subtends an angle of 44 deg. as shown in Fig. 4 (bottom). The supplementary angle made by planes tangent to the sphere at diametrically opposite points of contact with the original surface of the sample is 136 deg. This is also the angle made by opposite facets of a Vickers indentor.

Brinell hardness values calculated for ratios of L/D^2 of 5 and 30 are plotted in Fig. 3. The loads of 12.5 and 76 kg. are equivalent to loads of 500 and 3000 kg. on a 10-mm. ball. Lines representing Gray's (4) recent data for standard Brinell tests are drawn for comparison. The dotted lines again represent materials of intermediate work-hardening rates and roughly divide specimens having low and high Meyer n values. In the case of the $L/D^2 = 5$ data, the materials having low n values generally lie to the right and those having high n values to the left of the dotted line as in the cases of the $d/D = 0.1$ and $d/D = 0.2$ plots of Fig. 2. The relatively shallow penetration again accounts for this situation.

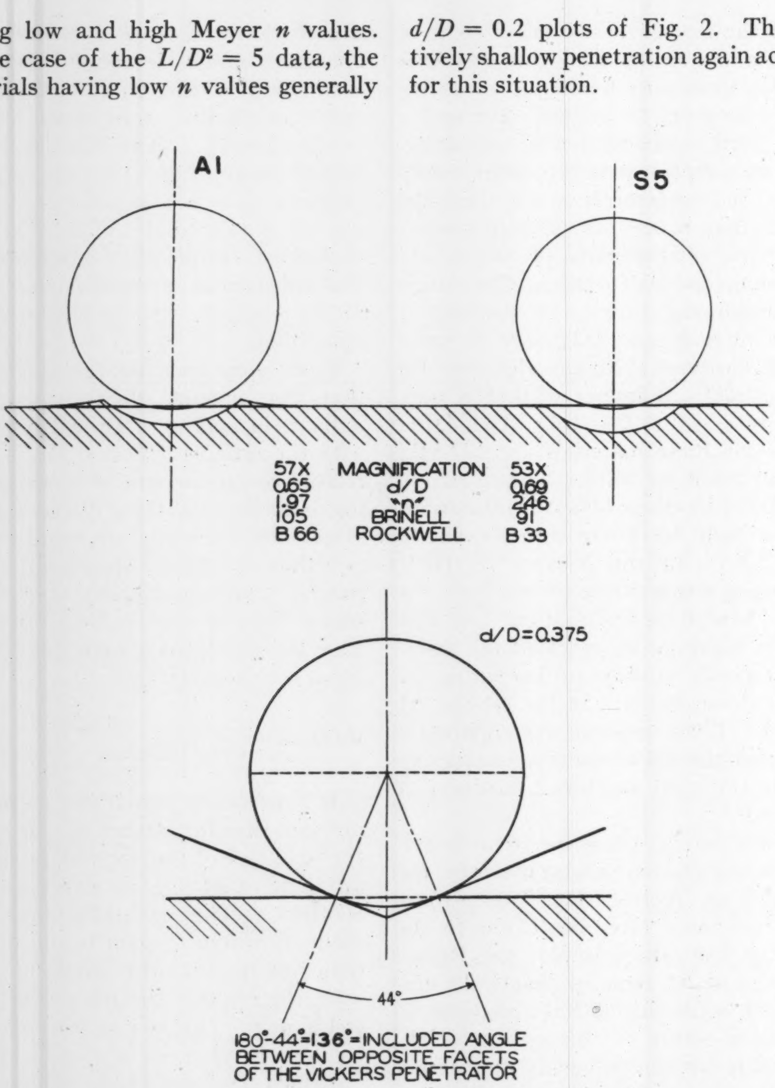
A1
S5
57X MAGNIFICATION 53X
0.65 d/D 0.69
1.97 "n" 2.46
105 BRINELL 91
B 66 ROCKWELL B 33
d/D=0.375
44°
180°-44°=136°=INCLUDED ANGLE BETWEEN OPPOSITE FACETS OF THE VICKERS PENETRATOR

FIG. 4.—*Top*—Sections Through Rockwell "B" Indentations in Materials of Similar Brinell Hardness but Different Work-Hardening Capacities.

NOTE.—Magnifications changed to approximately 18 and 17 in reproduction.

Bottom—Relationship Between the Vickers Indentation and the d/D Ratio for a Spherical Indentation.

In the case of the $L/D^2 = 30$ data, the positions of the specimens with respect to the dotted line are generally reversed.

This line represents Brinell values about 10 points higher than Gray's average line. However, it should be noted that the present Brinell data are inherently less accurate than those obtained under standard conditions with a 10-mm. ball and 500- or 3000-kg. load, therefore the results shown in Fig. 3 should be considered primarily as illustrative of the influence of work-hardening capacity on hardness conversions. The absolute values ordinarily used for conversion in this range are not in satisfactory agreement and several of the published relationships are more nearly in line with the present data than is Gray's relationship.

Effect of Contour of the Impression on Rockwell-Brinell Conversion Relationships:

The primary purpose of this program of investigation is correlation of DPH and the various Rockwell scales. Analyses of these relationships are complicated by the fact that Rockwell tests are based on a system of measurement that differs considerably from that of DPH or other Brinell type tests.

In order to illustrate this, special tests were made on materials A1 and S5. Standard Rockwell "B" tests were run, giving impression diameters of 1.032 mm. and 1.094 mm., respectively. The corresponding d/D ratios are 0.65 and 0.69; thus the impressions are quite comparable from the standpoint of a Brinell test, the calculated Brinell hardnesses being 105 and 91. In contrast to this similarity, the Rockwell "B" values are 66 and 33.

The samples were sectioned, photographed at 10 diameters, and enlarged to about 55 diameters. Exact tracings from these photographs are shown in Fig. 4, top. The circles were drawn in to represent to scale the $\frac{1}{16}$-in. ball penetrator in the position it occupied after the minor load was applied and before the major load was applied. The origi-

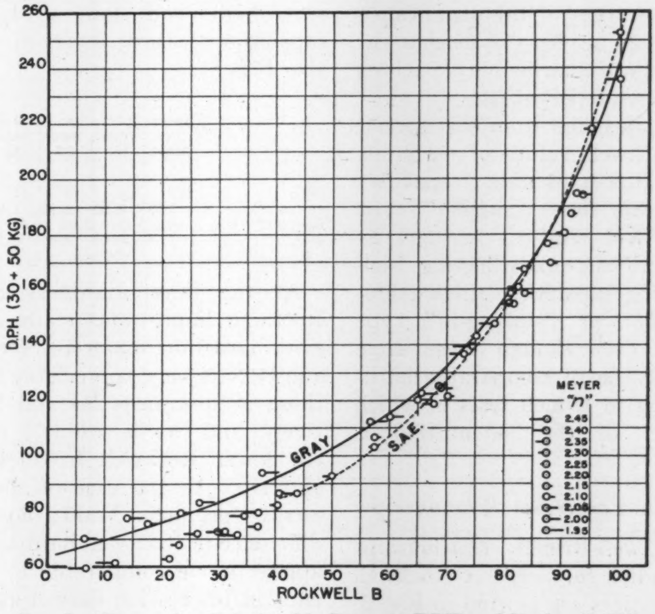

FIG. 5.—Conversion Relationship Between DPH and Rockwell "B".

nal surface of the sample is also indicated. Inspection of this figure reveals that while the impression diameters are comparable, the vertical penetration distances are not. Thus, the net movement of the Rockwell dial from "set" position to final position is 64 divisions (130 to 66) in testing specimen A1 and 97 divisions (130 to 33) in testing S5. Obviously the difference in contour between the two types of impressions is responsible for this behavior. The author's interpretation of the significance of contour types in terms of mechanical properties and work-hardening capacities has already been reported (3).

It can be concluded from this special test that Rockwell values of materials with low work-hardening capacities (these materials have "ridging" type impressions) will tend to be high as compared with materials having high work-hardening capacities (these materials have flat or "sinking-in" type impressions), even though these same materials may be of comparable hardness according to Brinell tests in which the diameter of the indentation is measured.

Rockwell-DPH Conversion Relationships:

The Rockwell-Brinell relationships were not studied further, but Rockwell "B" - DPH points are plotted in Fig. 5. The solid and dotted lines represent Gray's data and the relationship used in the 1942 Handbook of the Society for Automotive Engineers. It is interesting to note that under Rockwell "B" 80, Gray's curve represents materials with low *n* values, while the S.A.E. curve represents the high *n* values. It is clear that the influence of work-hardening capacity upon this relationship increases as the hardness decreases. This follows from the fact that the load of 100 kg. used in the Rockwell "B" test produces very large impressions in the softer materials. This magnifies the work-hardening effect to a much greater extent than in DPH tests or in Brinell tests. It will be remembered that in Brinell tests it is desirable to keep the ratio d/D within the limits of 0.2 to 0.6. In Figs. 2 and 4 (bottom), it was shown that the equivalent ratio for the Vickers penetrator is approximately 0.375.

In the following tabulation, it is shown that all Rockwell impressions under about "B" 65 have ratios of d/D over 0.6. The materials selected represent high and low work-hardening capacities at three levels of DPH (see also Fig. 5):

	Specimen MM1	Specimen C3	Specimen B3	Specimen A3	Specimen S5	Specimen D6
DPH	119	120	93	94	71	70
n	2.35	2.00	2.31	1.95	2.46	2.05
Rockwell "B"	68	65	50	38	33	7
d/D	0.59	0.65	0.66	0.76	0.69	0.84

At the lower hardness levels, the Rockwell impressions made in the low work-hardening materials, specimens A3 and D6, are very appreciably larger than those in specimens B3 and S5, respectively; and this is properly reflected in the lower Rockwell hardnesses of A3 and D6. In the case of specimen C3 *versus* specimen MM1, however, the difference in Rockwell hardness does not appear to be comparable to the difference in impression diameters. Further calculations give the following results:

	Specimen MM1	Specimen C3
(*a*) d/D	0.59	0.65
(*b*) d, mm. = measured impression diameter	0.94	1.03
(*c*) (130 − R "B" reading) = dial divisions	62	65
(*d*) ($c \times 0.002$) = penetration in mm.	0.124	0.130
(*e*) Penetration due to minor load, mm.	0.016	0.016
(*f*) ($d + e$) = total penetration, mm.	0.140	0.146
(*g*) Chord or diameter corresponding to (*f*), mm.	0.901	0.916
(*h*) (b/g) = measured/calculated impression diameter	1.04	1.12

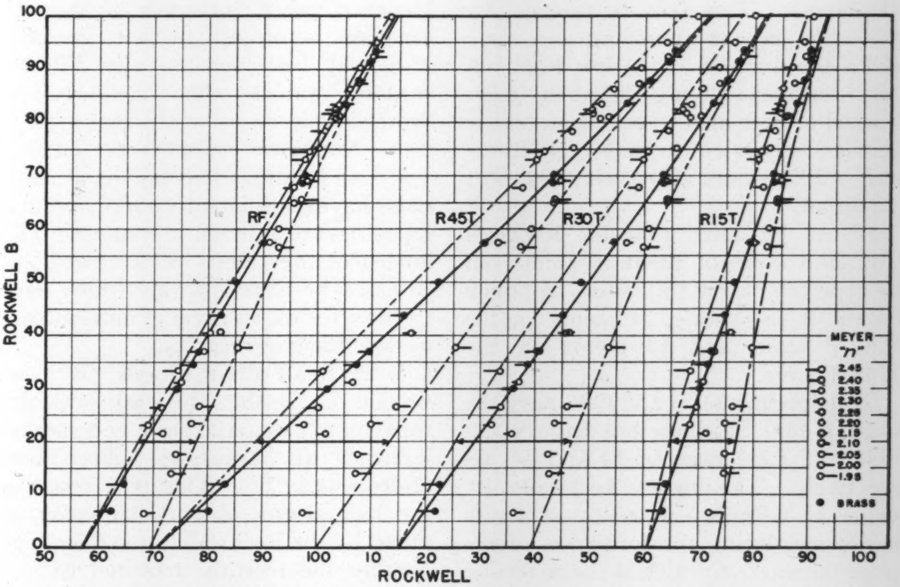

FIG. 6.—Conversion Relationship Between Rockwell "B" and Other Rockwell Scales Using a $\frac{1}{16}$-in. diameter Ball.

Thus, the measured diameter of the Rockwell "B" impression in MM1 is 4 per cent larger than calculated from the Rockwell hardness reading. The corresponding increase for C3 is 12 per cent, which means that the measured diameter of the Rockwell impression was very appreciably larger than the diameter calculated from the depth of penetration in the Rockwell test. This is the condition illustrated by A1 in Fig. 4; in fact, the comparison between MM1 and C3 at approximately the same Rockwell hardness is a corrollary to the comparison of S5 and A1 in Fig. 4, in which the impression diameters are comparable. In the case of both of the low n value materials, C3 and A1, the relatively small measured penetrations in the Rockwell tests result in high Rockwell "B" values. This geometrical factor tends to offset the direct effect of differences in work-hardening capacities, which were shown to be responsible for the major differences in "B" scale values between the pairs B3-A3 and S5-D6. When the impressions are very large, as in the case of S5 and D6, the direct effect of the work-hardening factor far outweighs the geometrical factor and a major difference in Rockwell "B" hardness results.

Rockwell - Rockwell Superficial Conversion Relationships:

The influence of work-hardening capacity is nowhere more in evidence than in the conversion relationships between the various Rockwell scales. The Rockwell "B", "F", 45-T, 30-T, and 15-T data are plotted in Fig. 6. The solid lines in this figure represent conversion relationships taken from Wilson Mechanical Instrument Co.'s Chart 38. The shaded points represent the brass test blocks and show that the hardness values for these blocks check the Wilson relationships very satisfactorily, with the possible exception of specimen B6 at 7 Rockwell "B".

It appears that the Wilson relationships apply to materials with high work-hardening capacity (annealed) in the low Rockwell "B" range, and to materials with low work-hardening capacity (cold worked) in the high Rockwell "B" range. This fails to provide for materials such as aluminum and some of its alloys which attain low Rockwell "B" hardnesses only in their cold-worked or hardened states, or austenitic stainless steels which have high Rockwell "B" values in their dead-soft condition.

It will be noted that for Rockwell "B" 20 a range of about 10 points can be expected on the "F" scale, 25 points on the R 45-T scale, 21 points on the R 30-T scale, and 12 points on the R 15-T scale.

Test specimens D3 and CB6 have the same R 15-T hardness but have Rockwell "B" hardnesses of 14 and 44, respectively. The higher Rockwell "B" value of CB6 is the result of strain hardening during the formation of the large indentation which is characteristic of the Rockwell "B" test as applied to soft materials. Specimen D3 was subjected to the same load of 100 kg. and the resulting indentation was even larger, but because of its low work-hardening capacity, the final Rockwell "B" hardness remained 30 points lower than that of specimen CB6.

CONCLUSIONS

1. The tendency of some metals to flow under load in the hardness tests makes it imperative to control the time of load application. Mild steel and ingot iron are among the materials which flow rapidly under load.

2. Comparison of DPH test results with Brinell hardness tests made with a $\frac{1}{16}$-in. diameter ball shows that the results are comparable when the ratio of Brinell impression diameter to ball diameter is about 0.375.

3. As a result of differences in work-hardening capacity, as measured by the Meyer n value, materials of identical hardness according to one hardness scale may have substantially different hardness values when measured by another scale or by a different type of test. Numerous examples are cited.

4. Ball hardness impressions in materials having different work-hardening capacities are different in contour. This influences the results of Rockwell and Brinell type tests in a way which tends to offset the direct effect of differences in work-hardening capacity.

5. In the DPH-Rockwell "B" conversion relationship, the work-hardening capacity of a material has a very marked influence at hardnesses below about 65-Rockwell "B". This is a result of the high degree of work-hardening induced by the Rockwell test in this range. Neither the recently reported data of F. H. Gray nor the S.A.E. conversions are adequate in this range.

6. The Wilson Mechanical Instrument Co.'s Chart No. 38 gives accurate results in converting the hardness of yellow brasses between any of the Rockwell or Rockwell Superficial scales using the $\frac{1}{16}$-in. diameter ball indentor.

7. Aluminum alloys and austenitic stainless steels are among the alloys having hardnesses in the Rockwell "B" range for which the Wilson chart No. 38 is not adequate for conversion between the various Rockwell scales.

REFERENCES

(1) Hugh O'Neill, "The Hardness of Metals and Its Measurement," The Sherwood Press, Cleveland, Ohio (1934).

(2) E. Meyer, "Untersuchungen über Harteprüfung und Härte," *Zeitschrift* des Vereines deutscher Ingenieure, Vol. 52, pp. 645, 740, 835 (1908).

(3) R. H. Heyer, "Analysis of the Brinell Hardness Test," *Proceedings* Am. Soc. Testing Mats., Vol. 37, Part II, p. 119 (1937).

(4) F. H. Gray, "Hardness Conversions for Metals Below 400 Diamond Pyramid Hardness," *Research Report R-94249-Q,* Westinghouse Research Laboratories, September 19, 1941.

DISCUSSION

MR. T. H. WICKENDEN[1] (*presented in written form*).—The author is to be congratulated on his excellent paper as it throws considerable light on the difficulties confronting anyone attempting to make an all-inclusive hardness conversion table, particularly in the lower range of hardness values. Mr. Heyer's tests were based on carefully selected conditions, testing machines checked against standards and the personal equation reduced to a minimum, but still considerable variation was encountered from three different factors. However, when hardness tests are made in the shop, additional variables creep in. The personal equation is a big factor; variations from the standardized test procedure is another; and condition of the testing machine is a third, so at best the conversion can give only approximate values.

The necessity for hardness conversion has been emphasized due to the war effort, as many plants are making parts designed and specified by engineering groups outside of their own organization, and using a different hardness system; hence a conversion table is necessary or new hardness equipment must be installed. Where a situation of this kind arises, a great deal of trouble could be avoided by making a hardness conversion chart for the two hardness systems involved to cover the specific parts, material, and heat treatment by arranging for cooperative tests with the principal, thereby taking care of many variables automatically. This is especially desirable in the lower range of hardness, and, further, a separate table should be made for each material, such as brass and bronze, steel, stainless steel, etc.

Mr. Heyer's analysis of the SAE diamond pyramid hardness *versus* Rockwell "B" values, as shown in Fig. 5, was done, I believe, without his knowledge of the material used in arriving at the SAE curve, and his deductions regarding the character of the materials used are correct, that is, materials capable of high work-hardness. The SAE tables are based entirely on steels of the constructional type as distinguished from tool steels, high-speed steel and stainless steels, and, further, these steels were in the annealed, normalized or heat-treated condition; no tests on cold-worked steels were included. In the lower end of the hardness range such steels as SAE 1015, 1010, and ingot iron were used in the annealed or normalized-and-drawn condition for making the determinations. In the higher range of hardness both plain carbon and alloy steels were used in various conditions of heat treatment. While the SAE table is indicated as approximate hardness conversions, an additional precaution is taken by showing numbers in italics for values beyond the practical range of usefulness of the machine, but included by a popular demand as a quick reference.

MR. W. E. INGERSON[2] (*by letter*).—In addition to providing much objective and useful data on hardness conversion,

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[1] Assistant Manager, Development and Research, The International Nickel Co., Inc., New York, N. Y.

[2] Bell Telephone Laboratories, Inc., New York, N. Y.

Mr. Heyer's very interesting paper provides another important step toward an understanding of what an indentation hardness test really measures.

An unfortunately large number of hardness conversion tables have been published without sufficient warning as to their limitations. This has led to a widely prevalent custom of regarding hardness as a fundamental property of a material, and expecting hardness conversion relations to be absolute, and the same for all materials. Many people have recognized, on the other hand, that the hardness test gives a result dependent upon a combination of properties, but it has not been clear which properties, or how they are combined.

It now appears that we may be in a position to say, at least as far as first order effects are concerned, what we are measuring when an indentation hardness test is made. First, let us recognize that a single hardness test compares to a single point on a stress-strain curve. Just as elongation under load, a single point on a stress-strain curve, is useful for specification and control, so is a single hardness test. But just as no one would be so fatuous as to assume that because two materials happen to show the same elongation under a certain load the entire stress-strain curves are identical, so no one should expect that because two materials show the same hardness on one particular scale they should show identical results for all loads on all types of penetrators.

Consider the following facts: Meyer found that the Brinell test could be systematized according to his well-known law

$$L = ad^n$$

Van Deusen, Shaw, and Davis[3] showed that the constant a is linearly related to tensile strength for a variety of alloys.

An analogous relation[4] has been found for the Rockwell ball test

$$\frac{(h)^m}{(d)} = \frac{C\Delta W}{Sd^2}$$

where C seems to be a fairly well defined universal constant, S the tensile strength, h the penetration depth, ΔW the difference between major and minor loads, and d the penetrator diameter. The constants n and m both are measures of work-hardening capacity. Thus it appears that conversion between the various Rockwell scales and between Brinell tests with different loading ratios are determined only by work-hardening capacity.

Now Mr. Heyer's work indicates that this is also true of the cross conversions Rockwell-Brinell-diamond pyramid hardness. Given a certain hardness determined by one method and a corresponding work-hardening capacity, we can state with confidence that the conversion relations will be the same as for any other material that has the same work-hardening capacity at the same hardness level.

Thus, to systematize the whole picture, it would seem that we require work-hardening capacity *versus* hardness relations. One way to present these is to obtain values of Meyer's constant n for various hardness. We may, for instance, plot n versus Bn for an alloy. Suppose that we have experimental hardness conversion relations for this alloy also. Then if on any other alloy we run a Meyer's analysis or the corresponding Peek-Ingerson analysis for Rockwell, and find the same value of n or m corresponding to the same hardness on any scale, we may be sure that the same hardness conversion relation will work. The accompanying Fig. 1 shows *m versus* Rockwell "B" relations derived from Wilson Chart 38, from the data recently taken on 70–30 brass and from data on 18 per cent chromium 8 per cent nickel stainless steel. One point taken on an aluminum alloy is also included. Note that the Wilson chart can be used to a good approximation on cartridge brass, but will be unsatisfactory for either stainless steel or aluminum.

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[3] H. N. Van Deusen, L. I. Shaw, and C. H. Davis, "Physical Properties and Methods of Test for Sheet Brass," *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part II, p. 173 (1927).

[4] R. L. Peek, Jr., and W. E. Ingerson, "Analysis of Rockwell Hardness Data," *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 1270 (1939).

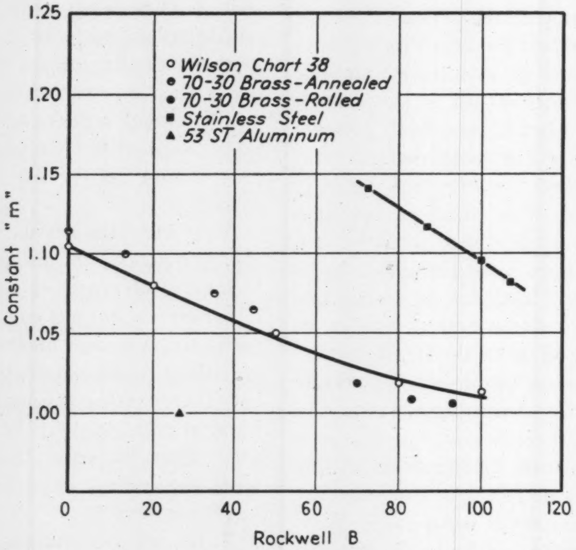

FIG. 1.—Relationship Between Constant *m versus* Rockwell "B" Hardness Derived from Wilson Chart 38.

Another line along which efforts in this field should be directed is that of using the hardness test as a research tool. Too often it is regarded as useful for inspection only. Yet it is one of the most direct ways of measuring work-hardening capacity, which is certainly related to the deep drawing and other forming characteristics of metals and alloys.

The influence of time in hardness testing is certainly important, and merits much more study. I hope Mr. Heyer's work will help stimulate the interest which the indentation hardness test merits, and that as time permits, additional research along these lines will be conducted.

MR. D. A. PAUL.[5]—I think Mr. Heyer is indeed to be congratulated on presenting us with some very interesting and worthwhile data.

We find in the case of aluminum alloys that when Rockwell scales having different ratios of load to square of ball diameter (L/D^2) are employed the conversion relation obtained for cold-worked metals is distinctly different from that obtained for annealed metals. The latter, of course, have greater work-hardening capacity than the former. When two scales having about the same ratio of L/D^2 are compared, however, one single relationship holds for both annealed and cold-worked materials.

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[5] Aluminum Research Laboratories, Aluminum Company of America, New Kensington, Pa.

If Mr. Heyer's results for the "F" scale, for which L/D^2 equals 19.9, are plotted against those for the "45-T" scale, for which L/D^2 equals 16.6, it will be seen that the points fall quite close to a straight line and that this line is not very different from that given by the Wilson Mechanical Instrument Co. Chart 38.

MR. J. J. KANTER.[6]—I should like to raise a point with regard to the diamond pyramid numbers. Presumably Mr. Heyer's chart is based upon a diamond pyramid with an included angle of 136 deg., which is correct for the Vickers test. Unfortunately there are a number of Vickers type indenters in use having included angle appreciably greater than 136 deg. and ranging anywhere from 140 to 155 deg.

Persons making tests with indenters having such a discrepancy in angle will find their results are considerably different from those obtained with the standard 136-deg. indenter, and inasmuch as so many domestically made off-angle indenters are in use, it would be highly desirable if the factor introduced by variation of included angle could be evaluated for use with Mr. Heyer's conversion chart.

MR. ROBERT H. HEYER[7] (*author's closure*).—The need for reliable hardness conversions is evident from the interest shown in this work. Two methods of attacking the problem of providing such conversions have been advanced in the discussions.

Mr. Wickenden recommends the establishment, by cooperative tests, of specialized conversions for specific materials or parts. This would insure substantial agreement between producers, fabricators, and purchasers using different types of hardness testing equipment. No doubt this principle is already in operation in many instances without official recognition. It is debatable to what extent the Society should act to recognize such special conversions as the one for cartridge case brass.

Mr. Ingerson's proposal to use work-hardening capacity as a basis for hardness conversions has the advantage of more general application. The determination of work-hardening capacity using Brinell type impressions is not a difficult undertaking; neither is it a routine matter. The corresponding Rockwell method may lend itself more readily to this purpose. However, experience with this test is as yet very limited, and while the experimental details may be less troublesome than in the Meyer test, it appears that the calculations are more so.

It is quite possible that hardness conversion relationships at different levels of work-hardening capacity, such as $n = 2.0, 2.1, 2.2, 2.3$, and 2.4, would be useful even though the "n" value were not determined for each hardness specimen. Sufficient data are available on the Meyer "n" values for important classes of materials so that a reasonable estimate could usually be made, but it would be necessary to know whether the material was hard rolled, annealed, or precipitation hardened, for example, because mechanical and heat treatment is often more important than composition in determining work-hardening capacity.

Of the two suggested procedures, Mr. Wickenden's proposal offers the most practical solution until time permits development of more general conversion relationships recognizing differences in work-hardening capacity.

Mr. Paul calls attention to the advantage of using the same ratio L/D^2 in ball-type tests, a principle which aids materially in eliminating the effect of differences in work-hardening capacities. Unfortunately there are very few combinations of hardness scales in which this ratio can be maintained.

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[6] Materials Research Engineer, Crane Co., Chicago, Ill.

[7] Metallurgist, The American Rolling Mill Co., Middletown, Ohio.

There is very little information available on correction factors for diamond indentors with nonstandard included angle. The Society specifies the standard angle of 136 deg. in the Standard Hardness Conversion Table for Cartridge Brass (Relationship Between Diamond Pyramid Hardness, Rockwell Hardness and Brinell Hardness) (E 33 - 42),[8] and all future standards in which Vickers or diamond pyramid hardness tests are included should be made to conform with this established practice.

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[8] 1942 Book of A.S.T.M. Standards, Part I, p. 925.

LONG-TIME VOLUME CHANGES OF PORTLAND CEMENT BARS

BY ALFRED H. WHITE[1] AND HAROLD S. KEMP[1]

SYNOPSIS

This paper presents a continuation of the studies reported in the A.S.T.M. *Proceedings* for 1911, 1914, and 1928.[2] There are available for study over 450 bars of known history which have been observed for periods ranging from 17 to 41 yr. The cements include a variety of commercial cements, and cements made in the laboratory whose composition differs in some cases quite widely from those found in commerce. A selection of 185 bars was made nearly 2 yr. ago for a special further study which involved alternations of the bars between the wet and dry states, with autoclave tests for some of the bars. Special attention was paid to the group carrying abnormally high magnesia.

All of the cements retain their property of expanding when placed in water and contracting when placed in air. Progressive elongation continues when the periods of immersion in water are longer than those of dryness. Mortar bars expand and contract less than those of neat cement, but show larger volume changes than would have been expected from the amount of cement they contain. The chemical composition of the cement and the percentage of mixing water influence the extent of the volume changes somewhat, but do not change their nature. The autoclave test did not cause excessive expansion of these old bars except where they contained larger amounts of magnesia than are permitted by the standard specifications. Not all of the bars containing high magnesia expanded in the autoclave test, since it apparently detects only free magnesia and not magnesia in combination.

The study of portland cement at the University of Michigan was commenced in 1899 under the direction of the late Prof. E. D. Campbell with the present senior author as a junior collaborator. Bauschinger in Munich had reported a few measurements on changes in length of cement bars in 1879, but the first systematic study of progressive changes in volume of cements was made at the University of Michigan and was published in 1906.[3] It was not realized at that time that hydrated cement would shrink when dry and expand when wet. The first presentation of this subject was made before this Society in 1911 by the senior author of the present paper, and further studies have appeared since that time.[2, 4] The chief value of the present paper comes from the long period of time covered by these studies.

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[1] Professor of Chemical Engineering, and Research Assistant, respectively, University of Michigan, Ann Arbor, Mich. The studies during the past year were financed in part by a grant from the Horace H. Rackham Fund of the University of Michigan.

[2] A. H. White, "Destruction of Cement Mortars and Concrete Through Expansion and Contraction," *Proceedings*, Am. Soc. Testing Mats., Vol. XI, p. 531 (1911).

A. H. White, "Volume Changes in Portland Cement and Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. XIV, Part II, p. 203 (1914).

A. H. White, "Volume Changes of Portland Cement as Affected by Chemical Composition and Aging," *Proceedings*, Am. Soc. Testing Mats., Vol. 28, Part II, p. 398 (1928).

[3] E. D. Campbell and A. H. White, "Some Conditions Influencing Constancy of Volume in Portland Cements," *Journal*, Am. Chemical Soc., Vol. 28, p. 1273 (1906).

[4] A. H. White, "Volume Changes in Concrete," *Transactions*, Internat. Eng. Congress, Volume on Materials of Engineering Construction, pp. 242-273, San Francisco (1915).

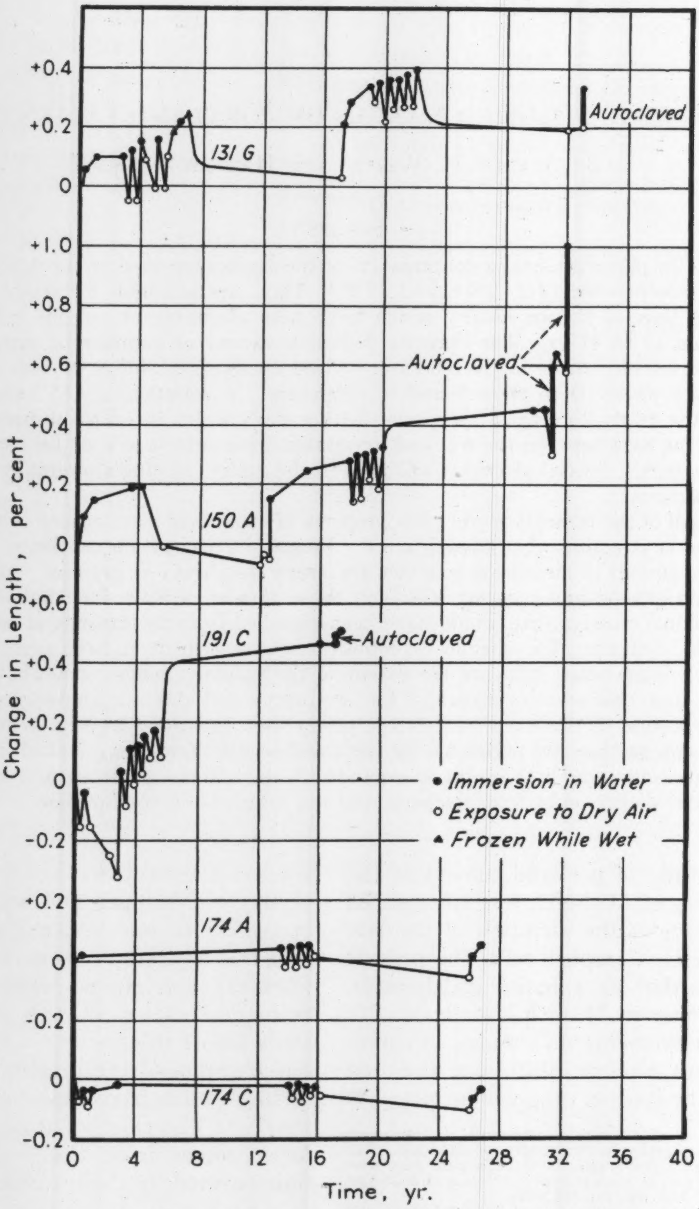

FIG. 1.—Change in Length of Bars of Commercial Neat Cements and of Cement Mortars when Immersed in Water and Dried in Air.

131 G, 150 A and 191 C are neat cements. 174 A and 174 C are 1:2 mortar.

VOLUME CHANGE OF COMMERCIAL CEMENTS AND CEMENT MORTARS

Commercial Cements:

The volume changes of bars of neat cement made from three commercial cements of acceptable quality are shown in Fig. 1. Bar 131 G was made from cement manufactured by one of the larger mills of the United States in 1908. It remained in water for 3 yr. reaching an apparently constant length with an expansion of 0.1 per cent. It was then removed from water and placed in dry air, then in water and again in air with four alternations as shown on the graph. Each time the bar was allowed to become thoroughly dry it shrank about 0.15 per cent, each time it was wet for several months the expansion was somewhat greater than it had been before, so that although after 3 yr. continuously in water its length had apparently stabilized at +0.10 per cent, its length after four alternations in four more years was +0.25 per cent. Between the sixth and seventh year the bar was frozen several times while completely saturated with water, without any measurable change in the length of the bar after it had been thawed out. This bar was then allowed to stand in air for 13 yr. and at the end of that period it had shrunk to approximately its initial length. When it was immersed in water again it expanded rapidly and after 2 yr. immersion was distinctly longer than at any previous time. It was then alternated five times between water and air during the next 2 yr. and after this series was completed its length when wet was +0.40 per cent. It was then left in dry air for 11 yr. and was then given a standard A.S.T.M. autoclave test.[5] The expansion was 0.13 per cent. Although the manipulation of the autoclave was according to A.S.T.M. methods, the procedure differed in that this bar was thoroughly dry when placed in the autoclave. The treatment with high-pressure steam did not expand this bar to the length it had previously attained when saturated with water. Our usual procedure would have been to have kept this bar in water to see whether it continued to elongate but one of the glass plates had become loosened during the autoclave treatment and so measurements were discontinued.

Another brand of commercial cement, cement 150 A, elongated to what was apparently a constant length of +0.20 per cent after 4 yr. in water, then contracted as it lay in air for the next 8 yr., expanded rapidly on being placed in water and then more slowly as it lay in water for 5 yr., contracted and expanded with 4 alternations between air and water during the next 2 yr. to a length of +0.32 per cent. After 11 yr. in water the length had grown to +0.46 per cent and on standing in dry air for 5 months it contracted to +0.30 per cent. It was then placed in the autoclave while dry, and, after the test, had expanded about 0.30 per cent. This was about 0.15 per cent more than it had shown when saturated with water. On leaving it in water for two months it continued to expand to +0.64 per cent, then contracted on 6 months in dry air to +0.58 per cent. It was then given a second autoclave test which caused it to expand an additional 0.43 per cent.

Cement 191 C is a high-alumina "Lumnite" cement stored in air after its first measurement with alternations between air and water as shown. On autoclaving after having been in water for 12 yr. it showed very little expansion.

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[5] **Tentative Method of Test for Autoclave Expansion of Portland Cement (C 151 - 40 T), 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 176.**

Cement Mortars:

Most of the bars of cement mortar were made in the laboratory with standard sand. They all show change in length with change from wet to dry conditions but in lesser degree than neat cements. The bars chosen for presentation are two made from a batch of commercial mortar which was being used as the top course of a pavement in Ann Arbor in 1914. The specifications called for a 1:2 mortar. One of these bars was kept immersed in water for nearly 14 yr. after it was removed from the mold. The other was kept in air for 15 days before being placed in water. It was then alternated as shown in Fig. 1. Bar 174 A which had its colloid more fully developed by long initial immersion in water expanded 0.10 per cent on the last alternation from dry air to water. Bar 174 C which was allowed to dry out for a time immediately after it was removed from the mold had a somewhat smaller expansion, 0.07 per cent, on its last immersion in water for 9 months.

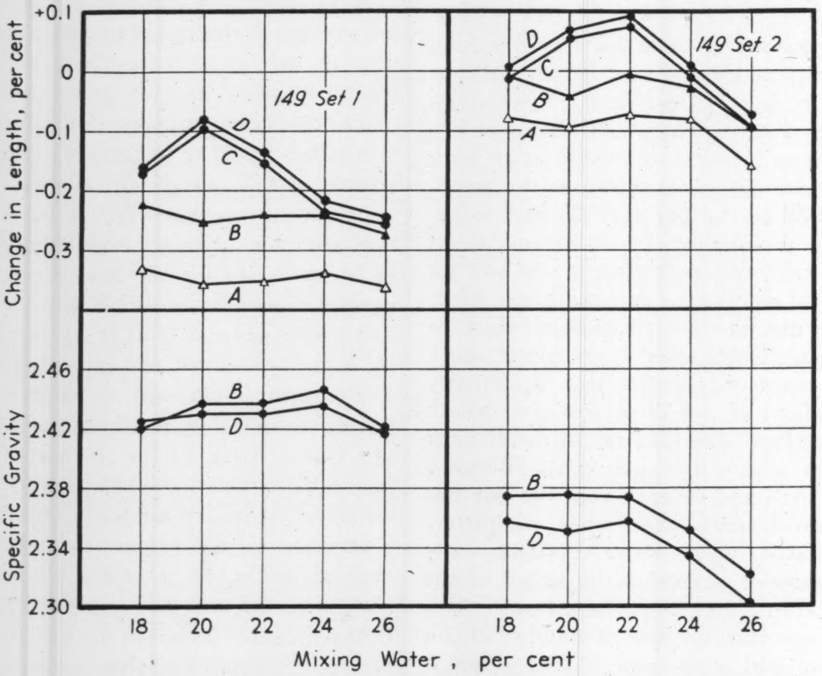

FIG. 2.—Effect of Variation in the Percentage of Mixing Water on the Changes in Length and Specific Gravity of Bars of Neat Cement.

The bars of set 1 were allowed to become dry as soon as they were removed from the mold. The bars of set 2 were allowed to dry out more slowly.

Curve *A* = after 4 yr. in air,
Curve *B* = after 27 yr. in water,
Curve *C* = immediately after autoclaving, and
Curve *D* = in water 2 months after autoclaving.

EFFECT OF VARYING PERCENTAGE OF MIXING WATER

A commercial cement was used and the percentage of mixing water was varied in five steps from 18 to 26 per cent. One set of five bars was placed at once in air and the second set was placed in a closed glass jar where it would not gain moisture and would lose it only slowly. These dry conditions were maintained for approximately 4 yr., and then all of the bars were placed in water for 27 yr., after which they were all given the standard autoclave test.

The variation in the percentage of mixing water gave a graded plasticity ranging from a very stiff mass with 18 per cent to a soft gel with 26 per cent of water. The differences in volume changes with varying water content were less than had been expected. The bars in set 1 which were placed in the air of the laboratory immediately after removal from the molds shrank rapidly and after 4 yr., their length differed in only a small degree from an average of -0.35 per cent as shown in curve *A* of Fig. 2. When these bars were placed in water they all gradually expanded about 0.10 per cent as shown in curve *B*. When given the autoclave test after 4 yr. in air and 27 yr. in water the expansions did differ, although they were small in all cases. The bar with 20 per cent mixing water expanded the most, 0.17 per cent, and those with 24 and 26 per cent mixing water expanded very little as shown in Fig. 2. These bars were left two months in water after being autoclaved and each one expanded slightly and to an almost equal degree.

The bars of set 2 were allowed to dry out quite slowly in a closed glass jar and so the cement was given a greater opportunity to react with the water. The resultant shrinkage when the bars were allowed to become thoroughly dry was distinctly less than in those of the first set and the elongation after 27 yr. in water was also less. When the bars were autoclaved, the maximum expansion again occurred in the bar with 20 per cent mixing water and the minimum expansion in the bar with 26 per cent water. The densities of these bars were determined after they had been immersed in water for 27 yr. and they offer a partial explanation of this behavior. The bars of set 2 showed less shrinkage after 4 yr. in dry air because of the initial storage in damp air, and they all showed a lower specific gravity than the bars of set 1 for the same reason. There was not much difference in the specific gravities of the bars with 18, 20 and 22 per cent water but a distinct decrease in specific gravity of the bars with 24 and 26 per cent mixing water. These two bars also showed almost no expansion after autoclaving. An apparently plausible explanation ascribes this to voids in those bars which were made with too much mixing water. This hypothesis will not explain the low expansion of the bars with 18 per cent of water, after the autoclave treatment.

EFFECT OF FINENESS OF CEMENT

An experiment to determine the effect of fineness of the cement powder was initiated in 1910. A commercial cement was sifted on a No. 200 sieve and the material passing and the residue remaining on the sieve were made into experimental bars. A separate sample was sifted on a No. 100 sieve and the coarser residue was made into a bar. These 3 bars and one made of the regular commercial cement were kept in water for 18 yr., then given 7 alternations of 60 days each between water and air, and then placed in water again. All of the bars show the characteristic changes in length between the wet and dry states. The regular cement with its mixture of relatively coarse and very fine particles shows the greatest variations in length and the residue left on the No. 100 sieve shows only about half as much, as might perhaps have been expected from the probable percentage of voids in the various fractions.

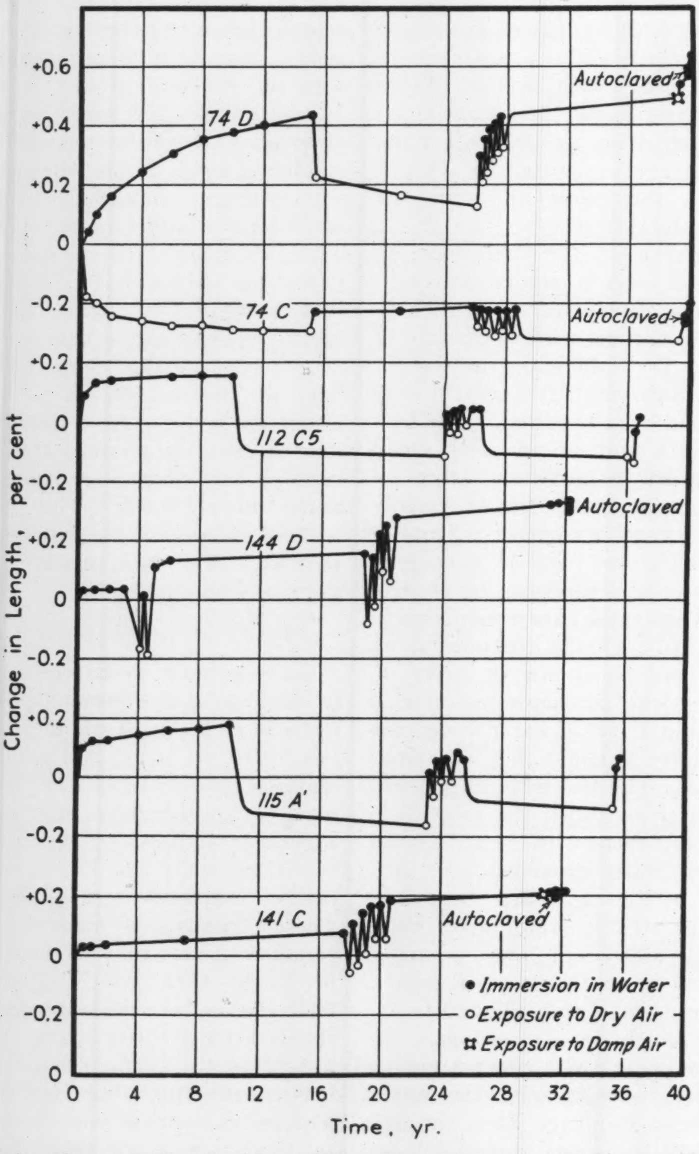

FIG. 3.—Changes in Length of Bars of Neat Cement Burned in a Laboratory Kiln.

CEMENTS BURNED IN LABORATORY KILN

The methods of manufacturing the laboratory cements as well as the chemical composition of some of the cements may be found in the papers[2] to which reference has already been made. Some of the cements were made from pure laboratory materials, and others were burned in the laboratory from materials in commercial use. The raw materials were ground wet in a ball mill. The temperatures of burning were carefully controlled and the cements all passed the soundness test unless there is a statement to the contrary. The following illustrations show the properties of some of these cements. The composition of the clinkers is given in Table I.

TABLE I.—COMPOSITION OF CEMENT CLINKERS.

	Cement 74	Cement 79	Cement 83	Cement 112	Cement 115	Cement 130	Cement 132	Cement 141	Cement 144
CONSTITUENTS, PER CENT									
SiO_2....	22.90	23.43	24.54	22.60	21.55	23.21	23.85	23.30	26.74
Al_2O_3....	7.64	7.77	6.80	11.15	9.09	6.54	6.78	3.08	2.77
Fe_2O_3...	2.81	2.67	3.17	0.35	2.56	0.56	1.95	6.23	0.14
CaO.....	59.59	58.92	63.10	65.90	63.01	62.25	59.15	64.40	70.06
MgO....	7.05	7.21	1.87	0	2.73	7.60	8.25	2.99	0
COMPOUNDS, PER CENT[a]									
C_3S.....	13.1	5.7	20.1	21.0	27.9	32.2	11.1	53.4	63.1
C_2S.....	55.7	62.9	55.2	49.0	40.7	42.2	56.0	26.5	29.1
C_3A.....	15.5	16.1	12.7	29.0	19.8	16.4	14.7	....	7.1
C_4AF....	8.5	8.1	9.6	1.0	7.8	1.7	5.9	14.7	0.4
MgO....	7.0	7.2	1.9	0	2.7	7.6	8.2	3.0	0
C_2F.....	....	....	....	....	....	....	....	2.4	....

[a] Calculated in the laboratory of the Portland Cement Assn.

Cement 115 was made from a ground mix sent us by one of the commercial cement mills. This was the only cement which was burned in a fixed kiln. The fresh cement passed the soundness test and set slowly enough so that no gypsum was added. Figure 3 shows that its volume changes were normal and smaller than the commercial cements given in Fig. 1.

Cement 112 was made from pure calcium carbonate, glass sand and kaolin so as to be free from magnesia and very low in iron. The cement was entirely sound when it was freshly ground and was mixed with 2 per cent calcium sulfate before it was made into an expansion bar. The volume changes as shown in Fig. 3 are normal and small in amount.

Cement 141 was made to determine the effect of low Al_2O_3 and high Fe_2O_3, with limestone, silica sand, and a clay modified by the addition of Fe_2O_3 as the raw materials. The analysis as given in Table I shows that tricalcium aluminate was absent and that there was 2 per cent of dicalcium ferrite present. The cement was entirely sound when freshly ground and bar 141 C was made immediately after grinding the fresh clinker. This bar has unusually small volume changes but shows the usual progressive expansion on alternations between air and water. It will be noted that this bar was in damp air for 9 yr. and continued its slow expansion. Exposure to damp air causes nearly as great expansion as immersion in water. It was next placed in water for 6 months and then autoclaved, contracting slightly after the autoclave treatment. After 2 months more in water it had expanded again to substantially its length before autoclaving.

Cement 144 was made in 1909 in an effort to approximate tricalcium silicate as closely as possible. After many failures it was found that a small amount of Al_2O_3 helped the clinkering process, and a sound cement was obtained by reburning a clinker containing 63.1 per cent tricalcium silicate, 29.1 dicalcium silicate, and 7.1 tricalcium aluminate. This cement shows normal and rather low volume changes, and slight contraction after being autoclaved when the bar was 32 yr. old, as shown in Fig. 3.

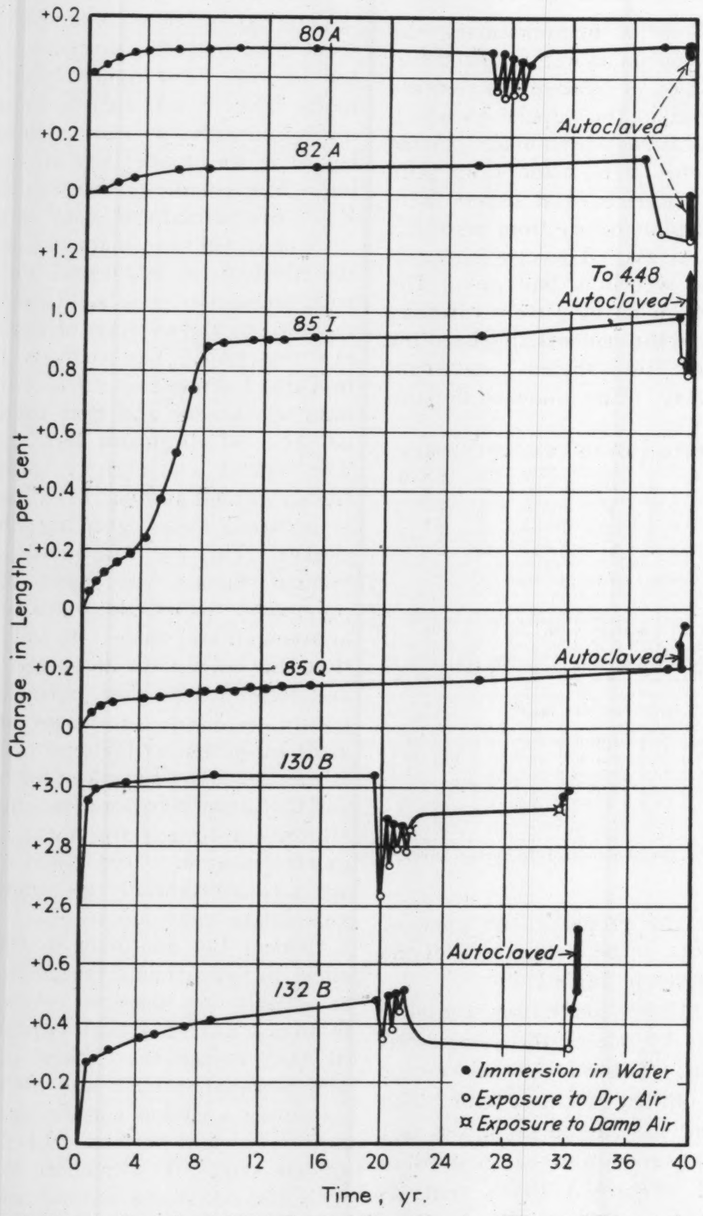

FIG. 4.—Variations in Length of Neat Cement Bars Showing Effect of Free and of Combined Magnesium Oxide.

EFFECT OF ADDED FREE MAGNESIA

The effect of adding magnesia to a sound commercial cement is shown in Fig. 4. The magnesia was prepared by burning magnesite in the laboratory kiln to the temperature usually used for burning portland cement, and then grinding it to the fineness of portland cement. Mixtures were made containing 1, 2, 3, and 4 per cent of this magnesia, with the commercial cement 85, and bars were made from each mixture. As a check another series of bars was prepared containing the same amount of MgO, added not as free MgO, but as the MgO in the magnesia cements 80 and 82 whose composition is given in Table II.

TABLE II.—COMPARISON OF COMPOSITION OF THE "ALL MAGNESIA" CEMENT WITH A NORMAL CEMENT.

	Cement 80 and 82		Cement 85	
	Percentage	Mols	Percentage	Mols
SiO_2	28.9	100.	24.5	100.
Al_2O_3	9.8	20.	6.8	16.4
Fe_2O_3	3.1	4.1	3.2	4.9
CaO	5.1	18.9	63.1	276.9
MgO	53.1	274.7	1.9	11.4

A report on one set of these bars was reported in the 1928 paper.[2] A duplicate set of bars was prepared as experiment 85. The life history of two bars of this latter experiment is shown in Fig. 4. Bar 85 I was made from this commercial cement with which 3 per cent of the finely ground hard-burned magnesite had been mixed. The bar expanded somewhat rapidly for the first 2 yr. but then elongated at an accelerated pace as it lay in water until at the end of 8 yr. its length was +0.890 per cent. It expanded extremely slowly as it became older and after 39 yr. in water its length was +0.993. It was then placed in dry air for 4 months and contracted a normal 0.12 per cent. An autoclave test at that time caused an elongation to 4.48 per cent, much too large to be shown on the graph. It lay in water for two months more and expanded an additional 1.35 so that its length became +5.83 per cent Bar 85 Q was made as a duplicate of 85 I with the single exception that the MgO was in the combined form of Cement 82 instead of in the free form. This bar showed only normal expansion as it lay in water for 40 yr. and on autoclaving at the end of that period.

Effect of Combined Magnesia—An "All Magnesia" Cement:

The similarity of CaO and MgO is great, and it is known that MgO combines with SiO_2 and Al_2O_3 to form slags. It was not known whether it could be made to combine completely in mixtures as basic as those normally found in portland cement. A commercial clay was accordingly mixed with pure magnesium carbonate and burned in the usual manner in the laboratory rotary kiln. It sintered at a temperature about that of a normal portland cement to give a dense, well-burned clinker, 80, which was ground and mixed with 1.5 per cent plaster of Paris. It required 21 per cent of water for normal consistency, took its initial set in 52 min. and its final set in 9 hr. It passed the normal boiling test for soundness, but did not give a strong pat. Only a small amount of cement was available for tension tests but ten briquettes of neat cement were made and stored in water. Two briquettes were broken after 28 days and showed a tensile strength of 36 and 49 psi., respectively. After 3 months two more showed 88 and 94 psi. At 1 yr. the figures were 96 and 131, and two briquettes broken after 2 yr. in water had a tensile strength of 157 and 166 psi. A second burning of this same mixture was made as cement 82. It gave a similar clinker which passed the soundness

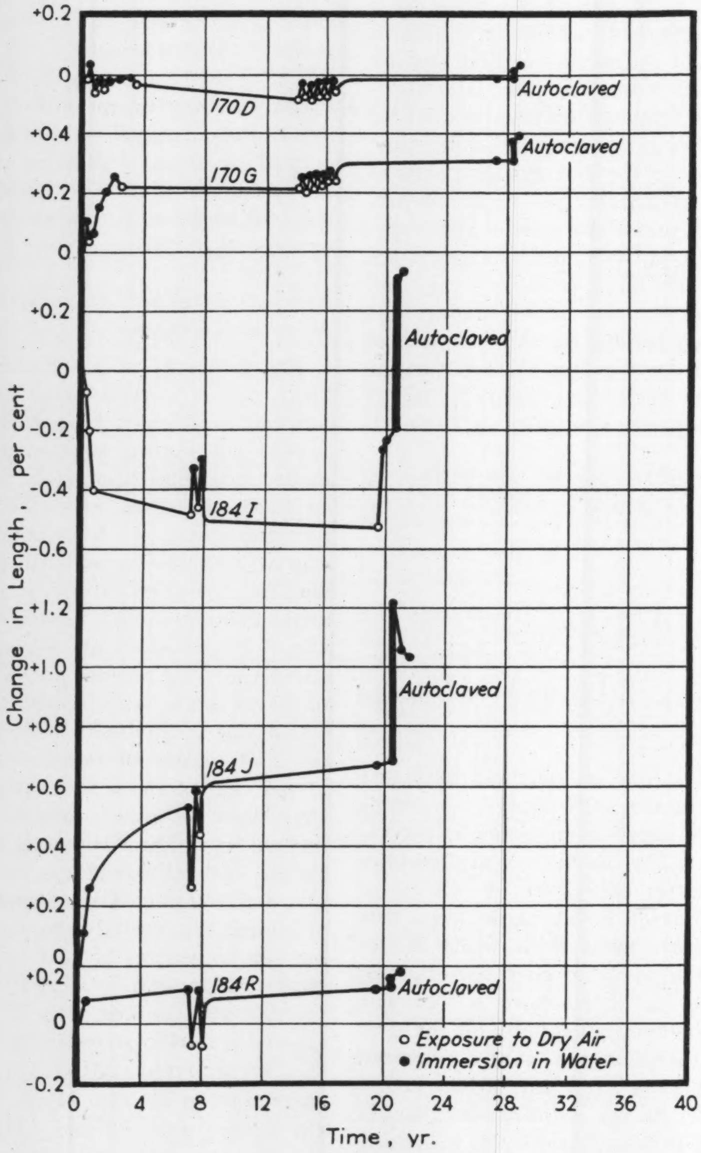

FIG. 5.—Variations in Length of Neat Bars of Natural Cements and of Portland Cement Mortar Containing Dolomitic Lime.

test. The behavior of one bar of each of these cements is shown in Fig. 4. Both of the bars 80 A and 82 A behave perfectly normally and show rather small changes in length when changed from dry to wet conditions and when autoclaved.

The composition of this cement is shown in Table II in terms of percentage and mols. It cannot be expressed in terms of compounds such as tricalcium silicate since it contains almost no lime. For comparison the composition of the commercial cement 85 is also given in Table II. If the CaO and MgO are added together the normal cement 85 has 288.3 mols RO to 100 of SiO_2 and the "all-magnesia" cement 80 has 293.6 mols RO to 100 of SiO_2. This "all-magnesia" cement complied with the old definition of portland cement in every particular unless it may be claimed that it was not "properly proportioned." No claim is made that this cement with 53.1 per cent MgO has value as a commercial product. It is presented as proof that large amounts of MgO may be brought into combination in a clinkered hydraulic cement.

THE AUTOCLAVE TEST ON OLD CEMENT BARS

The autoclave tests reported in this paper were made in the standard autoclave operated in accordance with the A.S.T.M. test procedure.[5] The tests differed in that they were made on old bars which had been exposed to different conditions of wetness and dryness. Most of the bars had been lying in water for rather long periods before being placed in the autoclave, but some were thoroughly dry and were taken at once to the autoclave from dry air. It is evident that when dried bars are placed in the autoclave there will be some normal expansion due to the hydration of the dried colloid and that an allowance should be made for this before calling an expansion abnormal. The expansion of the dried bars was not large as may be seen in bar 131 G of Fig. 1 and bar 82 A of Fig. 4. Bar 150 A of Fig. 1 is higher but not excessive.

The only bars whose expansion in the autoclave test was over 0.5 per cent were those to which hard-burned MgO had been added intentionally and those made from a natural cement known to be high in magnesia. Illustrations of this high expansion may be found in Fig. 4 where bar 85 I to which 3 per cent of hard-burned MgO had been added had expanded 4.48 per cent when measured after the autoclave treatment. The expansion is so large that the chart was altogether too small to record it and it is indicated by the number placed on the graph. Just above this curve in the same figure is the behavior of bar 82 A with 53.1 per cent combined MgO which was autoclaved under similar circumstances and showed only 0.16 per cent expansion. Illustrations of high expansion in a natural cement are shown in bars 184 I and J of Fig. 5.

It is quite evident that the autoclave test has detected free hard-burned MgO which had been ground finely, mixed with neat cement, and made into a bar only 1 in. square which had remained in water continuously for 40 yr. without hydration as instanced in Bar 85 I. It is true that the presence of free MgO had seemingly been shown by the large expansion of the bar during the first 8 yr. of its life, but the very slight progressive expansion during the next 30 yr. would have led to the belief that hydration had been substantially complete after 8 yr. in water. The autoclave test shows that only a part of the magnesia had hydrated and that the remainder was inactive because no water could force its way into the dense bar.

There have been a few cements which have shown an actual contraction on autoclaving, among them 141 C and 144 D of Fig. 3. They carry the highest percentages of tricalcium silicates and the lowest percentages of dicalcium silicates of all of our laboratory cements. They also contain the lowest percentage of Al_2O_3. These cements also show the minimum progressive expansion on long immersion in water. Cements 79 and 132 showed an abnormal expansion on long immersion which was attributed, in the 1928 paper, to free magnesia. The autoclave test indicates that free magnesia is not present in these cements in measurable amounts. The cements which show slow progressive expansion through many years are also those with high percentages of dicalcium silicate and it is possible that the progressive expansion may be due to the slow hydration of this constituent. There are too few different compositions available to make it wise to do more than call attention to this point.

Commercial cement 150 expanded from +0.30 to +0.59 per cent after being given the autoclave test while dry. This procedure was repeated 6 months later and it expanded from +0.58 to +1.01 per cent. The composition of this cement is not known.

MAGNESIA ADDED TO THE RAW MATERIALS AS CARBONATE

Cement 74 was made in the laboratory from clay and marl with an addition of magnesium carbonate sufficient to bring the MgO to 7.03 per cent in the clinker as shown in Table I. The lime in the mixture was rather low, only 13.1 per cent tricalcium silicate being calculated as present in the clinker. The slurry was not ground finely enough before burning and the fresh cement did not pass the soundness test. After storing the cement for 8 months it became sound, and bars 74 C and D were made from this stored cement. The life history of those two bars is shown in Fig. 3. Bar 74 D was kept continuously in water for 15 yr. with a progressive expansion to +0.44 per cent, which is not larger than some of the commercial cements of Fig. 1. It was then alternated between air, damp air, and water with a length after 40 yr. of +0.57 per cent. The bar was then removed from water and given a standard autoclave test but expanded only 0.02 per cent. On further storage in water it continued its expansion.

Bar 74 C was a duplicate of 74 D which was kept in dry air for the first 15 yr. of its life. It contracted in a normal manner and showed normal volume changes in alternations between air and water. In its fortieth year it was removed from dry air where it had lain for 10 yr., immersed in water two months, which caused an increase in length of 0.05 per cent and then given a standard autoclave test which caused an expansion of only 0.02 per cent. On storage in water for six months more it expanded 0.05 per cent to bring its length almost exactly to its initial measurement.

This cement contains 7.03 per cent MgO but the autoclave test does not indicate that it contains any free MgO. The slow expansion during 16 yr. in water to +0.44 per cent is larger than that of any of the other cements of Fig. 2. Two of these cements, 112 and 144, contain absolutely no magnesia and it will be noted that they show almost no progressive expansion after immersion in water for 2 yr. The other two cements 115 and 141 contain less than 3 per cent of magnesia and do show a slight continuous expansion while remaining in water. It may be that the rather large continuous expansion of cement 74 D indicates the slow continuous hydration of MgO, but the autoclave test does not indicate free magnesia. It might be argued that the magnesia had been completely hydrated by the long continued immersion in water and exposure to damp air. On the other hand it is difficult to see why bar 74 C did not expand in the autoclave since it had not shown any earlier expansion which should have come if any free magnesia had become hydrated prior to the autoclave test. The conclusion is that all of the 7.03 per cent MgO in this cement had become combined chemically.

MAGNESIA ADDED TO THE RAW MATERIALS AS SILICATE

The cements under discussion that had been made in the laboratory all had magnesia added to the raw material in the form of $MgCO_3$. It was decided to make a cement with the magnesia introduced in the raw mix as a silicate to see whether any of the magnesia would appear in the cement clinker as free MgO.

Cement 130 was made in the laboratory from kaolin, pure $CaCO_3$ and talc containing 28.5 per cent MgO. Its composition is shown in Table I. The total magnesia was 7.6 per cent. This cement was so low in Fe_2O_3 that it was not possible to obtain a clinker which did not contain free lime. The ground cement was aged 6 weeks but was still unsound when expansion bar 130 B was made. When placed in water it expanded very rapidly showing an elongation of 2.5 per cent in 7 days and 3.0 per cent in 1 yr. Figure 4 shows an expansion of 2.6 per cent as its first record. The volume changes after the first year are not abnormal and there is nothing to indicate that any of the talc has been converted to free MgO. This bar was not autoclaved, because it had expanded so much in its early history that it was feared that it would go to pieces in the autoclave.

Cement 132 was made as a continuation of this study. The raw materials were a limestone carrying only 1.2 per cent MgO, kaolin, and talc. The limestone contained enough Fe_2O_3 to make the mixture clinker more readily. The analysis of Table I shows 8.2 per cent of MgO, almost all of which had been added to the mixture in combination as talc. The cement was sound and set normally after an addition of 1.5 per cent of plaster of Paris. Bar 132 B showed normal expansion during its first year in water and then progressive expansion indicating perhaps a rather small amount of free magnesia. After 32 yr. it was autoclaved and expanded 0.21 per cent as shown in Fig. 4. Very little, and perhaps none, of the combined magnesia of the talc had been displaced to give free MgO.

Another cement made to test the behavior of talc as a constituent is cement 79 whose composition is given in Table I. The history of the bars of this cement are not shown graphically because of lack of space. It contained 7.2 per cent of MgO added to the raw mixture as talc. These bars showed only small expansion on autoclaving and the expansion while lying in water, although progressive, has not been extremely abnormal.

When the report on these cements was presented in 1928 it was stated that it was believed the continued slow and rather small expansion of these cements in water indicated the presence of free magnesia. The autoclave test has not confirmed this diagnosis, and there is no experimental evidence in the data as now developed to indicate that any of the magnesium oxide introduced into the mixture as talc has been converted to free MgO in the cement.

BEHAVIOR OF NATURAL CEMENT

An investigation of the volume changes in several of the commercial brands of natural cement was commenced in 1921. Bars of neat cement were made and alternated between the wet and dry states as shown in Fig. 5. Bar 184 R does not show any abnormalities. Bars 184 I and J were made from one of the natural cements known to be high in magnesia. Bar 184 I was kept dry for the greater part of the first 19 yr. of its life and showed a somewhat high contraction. After 1 yr. in water it expanded 0.32 per cent and when autoclaved it expanded 0.51 per cent. The companion bar 184 J was kept in water for the greater part of its life and after 19 yr. had expanded to +0.68 per cent. When autoclaved it expanded 0.53 per cent more. The bar was kept in water after autoclaving and during two months in water it contracted 0.19 per cent. This is the only bar in which such a contraction has been noted.

DOLOMITIC LIME AS AN ADMIXTURE IN MORTAR

Tests were commenced in 1913 with a mortar of one part of commercial cement and two parts of bank sand by volume. Other mortars were made with replacement of varying volumes of this same cement by an equal volume of commercial hydrated lime whose analysis showed 36.3 per cent MgO. The bars made without the substitution of any lime showed normal small changes in volume when alternately wet and dried. Bar 170 D was made with 10 per cent of the cement replaced with an equal volume of the hydrated lime. Bar 170 G was made with 3 volumes of cement, 7 volumes of hydrated lime, and 20 volumes of sand. The life history of these bars is shown in Fig. 5. The effect of the replacement of 10 per cent of the cement by hydrated lime is not detectable in 170 D. The replacement of 70 per cent of the cement by hydrated lime causes greater expansion so that at the end of 27 years bar G has a length of +0.31 while bar D has a length of −0.01. On autoclaving bar G expanded 0.07 per cent and bar D only 0.02 per cent. These variations are small but are believed to indicate free magnesia in the hydrated lime, since other bars of the set gave similar indications and a duplicate set made with high-calcium lime did not show this slow expansion on long-time immersion in water nor on autoclaving.

CONCLUSIONS

This study of the behavior of cement and mortar bars for periods as long as 40 yr. confirms the conclusions expressed in earlier papers. The variations in length when changed from water to air and from air to water not only persist but tend to become larger with successive alternations. This is probably due to the larger amount of colloid developed with time, and the filling of the voids so that there is less opportunity for internal expansion as the cement becomes hydrated more completely.

All of the cements show progressive expansion when alternated between air and water if the periods of immersion in water are fairly long. There is no indication that this progressive expansion can be prevented by changes in composition or methods of manufacture of the cement. The remedy lies in removing the cause and keeping the hardened cement structures in an environment where the moisture content is relatively constant.

The commercial cements available 30 yr. ago have shown larger volume changes than some of the cements made in the laboratory. The cements of today are made with much greater scientific knowledge and with much more perfect control of the manufacturing process than were those of a generation ago and it is quite possible that the commercial cements of today show smaller volume changes than these older cements.

Free magnesia in bars of neat cement makes itself evident by slow expansion on immersion in water. This expansion continues for several years and gradually ceases even when the bars are lying in water, probably because no more water can force its way into the bar. The autoclave test evidently detects this free magnesia which had not become hydrated even after immersion in water for 40 yr. Combined magnesia does not cause expansion in either water at room temperature or in the autoclave test. The autoclave test has not caused large expansion in any of the bars except those known to contain free magnesia.

DISCUSSION

Mr. F. O. Anderegg.[1]—Mr. White's cements were, of course, very much more coarsely ground than those of the present day, and the packing of the cement in the paste was much poorer, in all probability, than that we encounter now. Therefore, the volume changes were probably somewhat greater than would be obtained with cements of the present day under similar conditions.

Mr. H. H. Vaughan[2] (*presented in written form*).—This paper by Messrs. White and Kemp is most interesting as a continuation of that presented in 1928,[3] and it is, I think, safe to say that no such record of the behavior of a large number of cements under various conditions of storage, extending over so may years, has ever been made available. The experiments on alternating periods of storage in water and air, which were such a feature of the paper used in 1928, have been extended for a longer time, and the majority of the tests cover a sufficient number of years to justify the conclusion that no abnormal changes will subsequently develop.

While desiring to express sincerely to the authors my admiration of the paper as a whole, I feel that the types of cement selected for the investigation of the effect of MgO content were not representative of commercial practice. The only normal cement reported on in this connection is cement 85 containing 1.9 per cent MgO in the mix. Bar 85 I consisted of this cement with an addition of 3 per cent of hard-burned MgO and bar 85 Q of Cement 85 with the same amount of MgO in the combined form as existing in cement 82, which was constituted of 29 per cent SiO_2 and 53 per cent MgO. It is stated that the hard-burned MgO was ground to the fineness of portland cement, or say to a specific surface of 1600 to 1800. As the MgO grains found in practice are of 10 or 12-μ diameter, a 10-μ grain having a specific surface of 1900, it is evident that, apart from other considerations that will be referred to later, there is a decided difference between the action of MgO added in this form and that derived from the mix. This is illustrated in the accompanying Fig. 1 showing a comparison between a "laboratory cement group" containing varying MgO in mix, bar 79 C given in White's paper in 1928[3] containing 7.2 per cent MgO in the mix, and bars 83 D and 83 E also taken from the 1928 paper, in which 3 per cent and 4 per cent of hard-burned MgO had been added to normal cement 83. While the expansion in bar 83 D is slightly different from that of bar 83 E, it is of the same order, and it is important to note that at 10 yr. the expansion of 83 E is nearly ten times that of 79 C, and 83 D is over four times that of 79 C. The expansion of bar 79 C corresponds almost exactly with that of the 7 per cent MgO cements in a "Laboratory cement group" which confirms the accuracy of the 1928 report on its behavior.

In the case of the other cements employed as examples of high MgO content, the effect of the free CaO does not appear to have been given sufficient consideration and they were all laboratory-burned cements of unusual composition. Cement 74 made from clay, marl, and $MgCO_3$ is stated to have been sound after 8 months and was then used for bars 74 C in dry air and 74 D in water. Its MgO content was 7.03 per cent and while the expansion was not excessive, it was considerably greater than has been shown in other tests.

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[1] Consulting Specialist on Building Materials, Newark, Ohio.

[2] Consulting Engineer, Montreal, P.Q., Canada.

[3] A. H. White, "Volume Changes of Portland Cement as Affected by Chemical Composition and Aging," *Proceedings*, Am. Soc. Testing Mats., Vol. 28, Part II, p. 398 (1928).

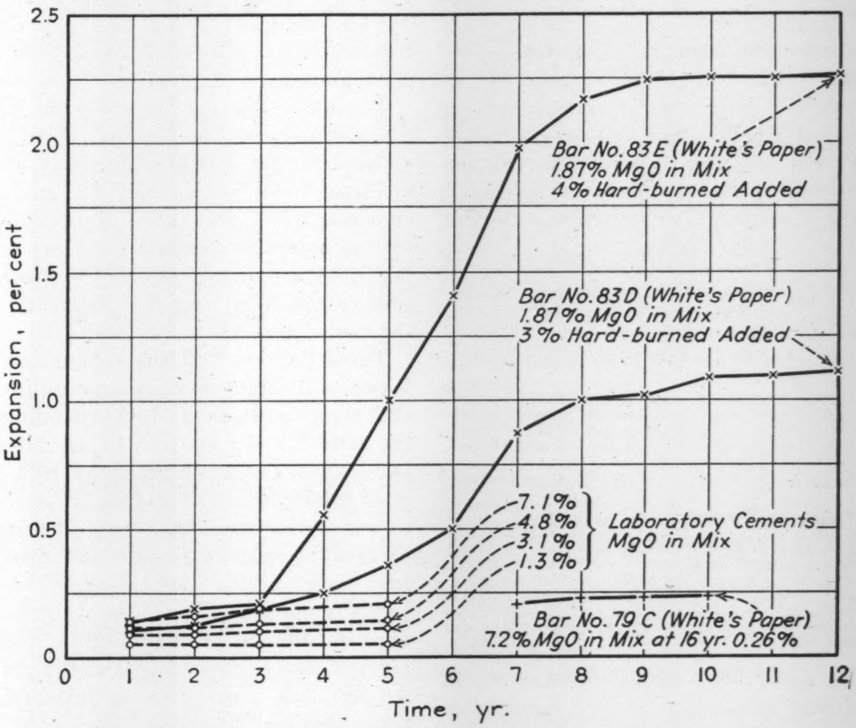

FIG. 1.—Comparison of Expansion Properties of Several Types of Cement.

Cement 130 made from kaolin, $CaCO_3$ and talc containing 28.5 per cent MgO, had an MgO content of 7.6 per cent but Fe_2O_3 so low that cement without free CaO could not be made. After aging 6 weeks, tests of this cement in water storage showed expansions of 2.5 per cent in 7 days and 3 per cent in 1 yr., evidently due to the high free CaO content.

I regret that Mr. White was unable to give us further data on bar 79 C reported in his 1928 paper; this was a normal cement with 7.2 per cent MgO in the mix and at 5 yr. showed an expansion almost exactly equal to that of the 7.0 per cent cements in a "laboratory group" and 0.26 per cent at 16 yr., entirely different from that of cements with MgO added.

I feel that in storage expansion tests it is desirable in so far as possible, to select cements of similar composition with one variable compound and for this reason a "laboratory cement group" is peculiarly valuable since, while the MgO contents varied from 1.3 per cent to 9.8 per cent, the free CaO contents were in all cases 0.3 per cent or less. In these cements in which the MgO was derived from the mix, the expansion both at 1 and 5 yr. was remarkably constant in its relation to the MgO content up to 7 per cent or 8 per cent above which there was a tendency to increased expansion. When cements contain higher free CaO, the expansions are more irregular, as in some cases 0.4 per cent may introduce a distinct effect while in others 0.7 per cent or more makes little difference. An analysis of 66 cements in Lerch's paper[4] (those with over 1.15 per cent MgO) shows 0.019 per cent expansion per 1 per cent MgO content at 1 yr. and 0.024 per cent at 2 yr. when corrected for constant expansion independent of MgO. Thirty-two of these cements with less than 1 per cent free CaO show 0.018 per cent expansion at 1 yr., and 0.024 per cent at 2 yr., while a new "laboratory cement group" shows 0.020 and 0.025 per cent, respectively, figures closely corresponding to those in the accompanying Table I, when additional expansion due to free CaO is allowed for.

TABLE I.

Content, per cent	Corrected Expansion			Expansion per 1 per cent MgO		
	1 yr.	1 to 5 yr.	5 yr.	1 yr.	1 to 5 yr.	5 yr.
1.3	0.025	0.009	0.034	0.0192	0.0069	0.0261
3.1	0.064	0.021	0.083	0.0206	0.0068	0.0268
4.8	0.091	0.032	0.122	0.0189	0.0067	0.0255
7.1	0.127	0.056	0.183	0.0180	0.0079	0.0260
Average				0.0192	0.0071	0.0261

Figures corrected for constant expansion of 0.015 per cent, that is, independent of MgO content. Note reduction in this amount decreases expansion per 1 per cent of MgO content as latter increases.

Expansion per 1 per cent MgO content is measure of amount of MgO expanding in the ratio 1 to 1.3. The proportion of the MgO content expanding in this ratio is therefore: At 1 yr.—5.76 per cent. At 5 yr.—7.87 per cent. From 1 to 5 yr.—2.13 per cent.

In the new "laboratory cement group" free CaO was intentionally varied and in this series it can be shown that while there is a considerable difference in the expansion caused by any given free CaO content, there is distinct evidence that the expansions caused by the two compounds MgO and CaO are simply additive; in fact the higher expansions per 1 per cent free CaO occur in the cements with 1 to 1.6 per cent MgO and the lower in those with 4.8 to 5.3 per cent MgO. On the average the added expansion at 1 yr. in water storage is from 0.07 to 0.10 per cent per 1 per cent free CaO or about four times that caused by an equal amount of MgO.

The accompanying Table I taken from a "laboratory cement group" gives the expansion that actually develops at 1 yr., from 1 to 5 yr., and at 5 yr., and the expansion per 1 per cent of MgO content for the cements from 1.3 to 7.1 per cent MgO. The footnote shows the proportion of the MgO content expanding in the ratio 1 to 1.3.

Table I shows that the expansion per 1 per cent MgO is singularly uniform, that the proportion of MgO expanding in the ratio 1 to 1.3 up to 5 yr. is under 9 per cent and that the expansion at 1 yr. is 73 per cent and that from 1 to 5 yr. 27 per cent of that at 5 yr. This indicates a rapid reduction in the rate of expansion as the age increase which logically follows if the hydration takes place on the surface of the grain and is independent of the volume of the grain. To test this, finely crushed fused MgO was obtained from the Norton Co., was separated into groups of particle sizes by the Haultain Infrasizer, and the average particle size in each group as determined by the turbidimeter was as follows:

Haultain Infrasizer	over 56 μ	56 to 40 μ	40 to 28 μ	28 to 20 μ	20 to 14 μ	less than 10 μ
Turbidimeter	65 μ	44.5μ	31 μ	22 μ	16.8μ	3.8 μ

One gram of each sample was placed in a weighed crucible, covered with water, and then placed in a desiccator from which the calcium chloride had been

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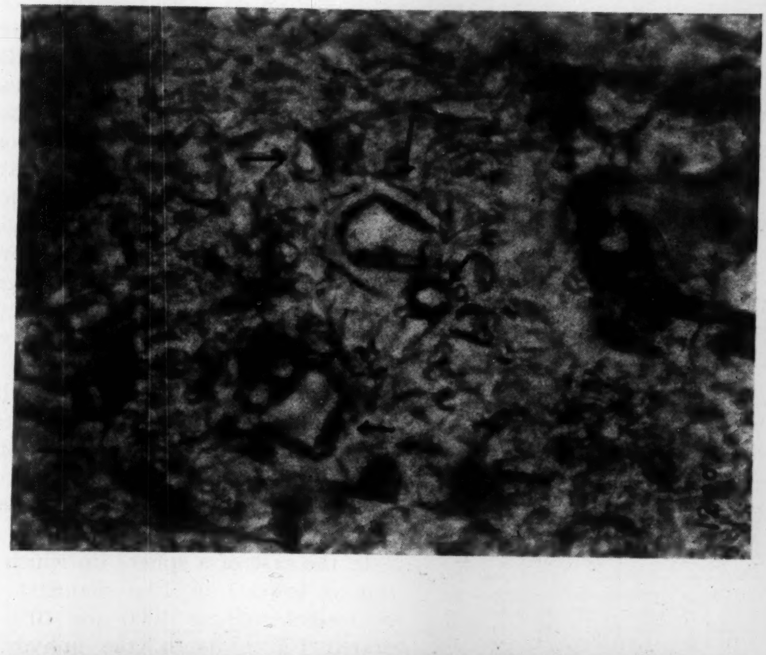
[4] William Lerch and W. C. Taylor, "Some Effects of Heat Treatment of Portland Cement Clinker," *Paper No. 33*, Portland Cement Assn. Fellowship at the National Bureau of Standards, July, 1937.



(a) Fresh-water storage 7 yr.

(b) Air storage 8 yr.

FIG. 2.—Photomicrographs of Cement in Water and Air Storage (× 1200).

removed. Samples were dried in the oven to constant weight. Increase in weight was taken at 5, 7, 10, and 14 days.

The depth of hydration was from 0.32 to 0.41 μ at 5 days and averaged 0.9 μ in depth in 14 days, the figures for the various groups being as follows:

Grain size..	65 μ	44 μ	31 μ	22 μ	17 μ	3.8 μ
Depth hydrated...	1.05 μ	0.95 μ	0.7 μ	0.65 μ	1.25 μ	0.8 μ

Although the grains were irregular in form and varied 5000 to 1 in volume, it is evident that the depth was practically constant and varied directly with the time.

TABLE II.

Storage and Time	Diameter of Grain, μ		Original Diameter, μ	Depth Hydrated, μ	Hydration, per cent		Time, 100 per cent Hydration, yr.
	Over-all Hydrated	Unhydrated Portion			Diameter	Volume	
Water, 7 yr.	12.35	6.25	10.0	1.88	38	76	19
[a]	9.55	3.75	7.5	1.88	51	88	14
[a]	8.85	3.75	7.0	1.6	47	85	13
	5.75	1.57	4.5	1.46	65	91	11
Air, 8 yr.	13.2	8.6	11.5	1.45	23	54	35
	11.45	6.5	9.3	1.40	30	66	27
	10.4	7.7	9.1	0.72	16	40	50
	4.15	2.2	3.4	0.60	35	72	23
	3.7	1.8	3.0	0.60	40	78	20

The cements from which the photographs shown in Fig. 2 were taken contained about 6.7 per cent MgO and probably an appreciable amount of free CaO with a corresponding expansion up to 3 or 4 yr., considerably higher than in the "Laboratory cement group."

[a] Same grains alternative determinations.

This experiment determines the rate of hydration in water at normal temperature. While this rate varies with the condition under which hydration takes place, photomicrographs of cement in water and air storage for 7 or 8 yr. also indicate that the hydration takes place at the surface. These photomicrographs are shown in the accompanying Fig. 2.

The accompanying Table II, giving the results of measurements from Fig. 2, indicates that both in water and air storage the depth of hydration is greater in the larger grains than in the smaller. This may be due to the increased volume hydrated in the larger grains per unit of depth hydrated, resulting in greater pressure on the surrounding material, thus increasing the rate of hydration, or to the greater expansion causing strains which permit a freer access of water vapor. This latter explanation is supported by the depth of hydration being roughly proportional to the square root of the diameter of the grain, the same function as was found to govern the relation between the size of the grain and the point at which fissuring occurred in the experiments on cements with additions of fused MgO.[5]

The maximum variation in depth of hydration is, however, only 2.6 to 1 with a variation in the volumes of 50 to 1, so that a uniform depth of hydration per unit of time is reasonably established.

In the case of a sphere (in which the rate is lowest) of 10-μ diameter and equivalent volume 1000 (or 10^3) hydrated 1 μ in depth, the unhydrated diameter is 8 μ and the unhydrated volume 512 (8^3). For various depths of hydration the relation is as follows:

Depth of hydration, percentage of diameter	5	10	15	20	30
Unhydrated volume, percentage of original	73	51	35	22	6

The calculations are exceedingly simple, and by a slight modification the original diameter of a partially hydrated grain can be determined when the over-all hydrated diameter and the unhydrated diameter are known. This method was used in Table II. The results lead, however, to one important conclusion. The expansion is proportional to the volume hydrated, so that it is evident that in the case of grains hydrated 40 to 50 per cent in diameter such as the larger grains in water storage in Table II, 75 to 85 per cent of the total expansion has already taken place, and there is no possibility of any large amount of delayed expansion developing. That idea has arisen from the theory that 5 per cent MgO will ultimately cause 1.5 per cent expansion, ignoring the fact that only a small proportion of the MgO content actually causes expansion and that most of that has already taken place within the periods covered by existing tests.

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[5] H. H. Vaughan, "Expansions in the Autoclave Caused by Magnesia," *Rock Products*, April, 1940, Vol. 43, No. 4, pp. 48 to 50; "Autoclave Expansion Experiments," *Rock Products*, June, 1941, Vol. 44, No. 6, pp. 48 to 90.

The large proportion of the expansion that occurs up to 1 yr. is only partly caused by the above reasons. The MgO grains vary considerably in size and consequently have different volumetric rates of hydration and their hydration is completed in different times. Their volumetric hydration curves are however similar in form and it is possible to analyze the expansion curves obtained by experiment to obtain a rough idea of the time in which different portions of the MgO content hydrate and their relative proportions.

The accompanying Fig. 3 shows the actual expansion curve of the 7 per cent MgO cement in a "laboratory cement group" water storage and also the curves of the MgO expansion resulting from grains hydrating at ages of 3 months, 1 yr. and 20 yr., respectively. Similar curves have been worked out for the 1.3, 3.1, and 4.8 per cent MgO contents, and while there is no claim that they are unique, they indicate reasonably clearly what may be called the short and long term components causing the expansion. As might be expected from the method employed, these are reasonably consistent for the four cements, the final figure being about 0.33 per cent expansion per 1 per cent MgO content corresponding to 11 per cent of the MgO

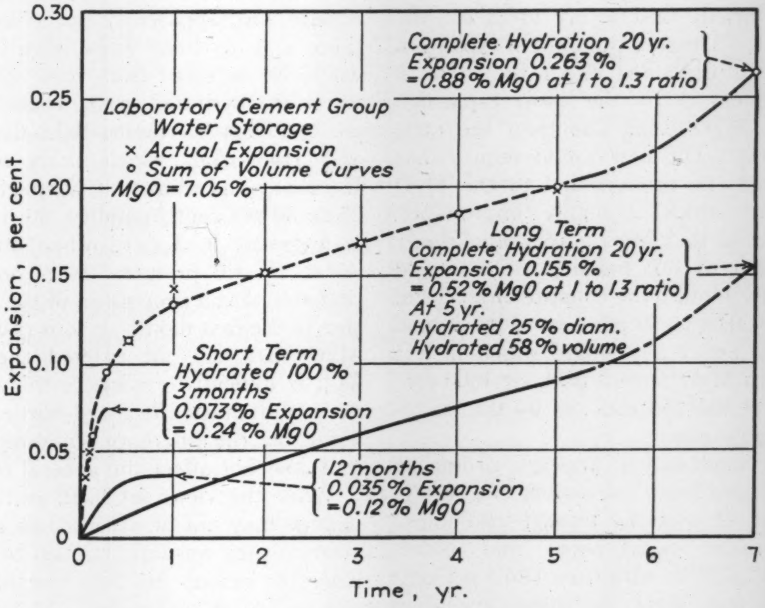

FIG. 3.—Expansion of Cements Stored in Water and Those Hydrated at Several Ages.

expanding in the ratio of 1 to 1.3 as compared with 8.7 per cent at 5 yr. For the 7 per cent MgO cement the final expansion is 0.263 per cent corresponding almost exactly with White's 79 C at 16 yr.

Two important facts appear in this discussion—the uniformity of the expansion caused by the MgO, and the comparatively small proportion causing expansion. Taken together their explanation is not obvious and to save time I will briefly outline my ideas on the subject. The small amount of expansion might be explained either by voids in the cement or by the linear expansion of the MgO being less than the ratio 1 to 1.3. The first would require that the voids be proportional to the MgO content, which is highly improbable; the second that MgO grains should only expand 1 to 1.03 linearly. This would not only require the collapse of a crystal of $Mg(OH)_2$ to 50 per cent of its normal volume under a pressure which in a 5 per cent MgO cement is about 4000 psi., but it is also contradicted by the microscopical evidence.

One explanation appears promising based upon Lerch's discovery of a certain amount of what he termed insolubles.[6] In so-called "rapid-cooled" and "plant-cooled" clinker, with from 1 to 5 per cent MgO, the MgO in these insolubles amounted to about 9 per cent of the MgO in the cement and was independent of the glass content. It was considerably greater in the "slow-cooled" clinker, but this fact is only of academic interest, as while that method of cooling can be carried on in a laboratory, it cannot occur in cement manufacture. It, however, indicates that MgO will crystallize out from the melt while the range of temperature at which that action may occur persists. Photomicrographs of these crystals show that they correspond in size with those shown as partially hydrated in storage specimens, and the possibility that they account for the longer term expansions is quite reasonable. The MgO that is not taken into solution in the melt will have been heated to considerably over 1000 C. and converted to the dense-burned state and remain in that unchanged up to at least 1650 C. Its crystals are exceedingly minute but apparently form aggregations and hydrate more slowly than would be expected from their size. In water the hydrated grain would be 0.5 per cent less in volume than the MgO and H_2O from which it is formed. If wetted and allowed to dry in air of 25 to 30 per cent humidity, 90 per cent is hydrated in 5 days when absorbed water is still present. It is, however, probable that a proportion of the expansion in the first month or so is caused by MgO that has not entered the melt, as it is difficult to reconcile the various rates of hydration for hard-burned MgO alone, but the difference is not important and does not affect the general results.

While the views set forth in this discussion may not be accepted as entirely correct, they certainly explain to a considerable extent the behavior of those cements in this paper to which I have referred. The abnormal expansion that occurs when hard-burned MgO is added is due to the expansion caused by the addition being about ten times greater than that caused by the same amount derived from the mix and by the difference in the size of the grains. If those in the addition at all resemble those in cement ground, their size distribution will vary considerably from those causing expansion in normal cement. It should be noted that while a given volume of 5-μ grains will cause three time the expansion of the same volume of 20-μ grains when both are hydrated 2 μ in depth, the increase in volume for one 20-μ grain is over twenty times that for each 5-μ grain and the tendency to fissuring is correspondingly greater. These two facts explain the rapid expansion between 2 and 8 yr. in bar 85 C while the residual expansion and that in the autoclave are caused by some of the larger but better protected grains still remaining only partially hydrated, as might readily be the case if retained even on a 325-mesh sieve.

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[6] William Lerch, "Approximate Glass Content of Commercial Portland Cement Clinker," *Journal of Research*, Nat. Bureau Standards, Vol. 20, No. 1, January 1938, p. 77 (*RP1060*).

The amount of expansion in a normally manufactured cement from a given MgO content has been determined. It is moderate in amount and its effect is practically completed within the period covered by the existing tests such as those of a "laboratory cement group" and by Bates.[7]

As regards behavior in service, I quote a statement made by Dr. Ary Torres, Director of the Institution of Technical Research of São Paulo, Brazil, in the introduction to his specifications for portland cement in June 1935: "In the case of MgO, we are increasing by 1 per cent the limit adopted by the Americans and others because the national cement of older manufacture which contained a percentage of magnesia approximating this quantity has been employed for many years and yet no concrete case has been discovered to our knowledge that would authorize us to suspect its behavior in construction."

The cements he refers to were manufactured from 1927 to 1933 and have, therefore, now been in service up to 15 yr. Recent examination has confirmed Dr. Ary Torres' statement as to their stability.

MR. ALFRED H. WHITE[8] (*author's closure, by letter*).—The careful studies of Mr. Vaughan have added materially to our knowledge of the effect of magnesia in portland cement. I regret that I cannot furnish more information from our own tests in further discussion of some of the points he raises.

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[7] P. H. Bates, "Properties of Portland Cement Having a High Magnesia Content," *Technologic Paper No. 102*, Nat. Bureau Standards (1918).

[8] Professor of Chemical Engineering, University of Michigan, Ann Arbor, Mich.

THEORY AND MEASUREMENT OF PLASTICITY AND WORKABILITY OF MORTARS[1]

By Paul S. Roller[2]

Synopsis

The plasticity of mortars is measured on the basis of a stress-strain relation that previously had been formulated and applied to various dispersions. Two constants are determined, coefficient of plasticity, π, and yield value, p_0, only the latter depending on the mixing water content.

Plasticity is defined in terms of ease of deformation and interpreted as one of the factors of workability of which four are discussed. The other three factors are: tendency to rupture during plastic flow, tendency to suspension of plastic flow, and water retention. π is shown to determine plasticity, and to be a good measure of over-all workability.

Water retention, as one of the factors of workability, was measured directly and with p_0 at 16.7 per cent mixing water as an index. It was found to be well correlated with π for a series of 1:3 mortars of portland cement with added mineral powders. Relative to these materials at the same plasticity, the water retention of 1:3 mortars of several masonry cements and a slag cement seemed to be low.

Theoretical factors involved in plasticity and workability are discussed.

Previously the author interpreted plasticity on the basis of a simple stress-strain relationship, discussed the theory, and dealt with a variety of dispersions of liquid and solid, such as clay and water, etc.[3] Although mortars were included as examples, in this paper they are considered more specifically and extensively.

Plastic Flow and Plasticity

Among physicists and metallurgists dealing with metals, plastic flow is defined as the irreversible deformation of a metal under stress. There is no reason why the same definition should not also apply to dispersions, such as mortars, clay pastes, lime putties, greases, etc., as under stress these also undergo a deformation that is irreversible and permanent. The author's interpretation of plastic flow of dispersions is therefore based upon this property. It is judged to hold accurately, except for high-speed deformations, when viscous resistance and the time element have then to be taken into account.

Because of the greater stability and more extended use of dispersions under compression, the stresses considered are compressive ones, and the deformations are given by the height of a cylindrical specimen, h, as a function of the pressure, p.

The relationship that was found to

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[1] Published by permission of the Director, Bureau of Mines, U. S. Department of the Interior.

[2] Physical Chemist, Bureau of Mines, U. S. Department of the Interior, Eastern Experiment Station, College Park, Md.

[3] P. S. Roller, "Plastic Flow of Dispersions and a New Approach to the Study of Plasticity," *Journal of Physical Chemistry*, Vol. 43, p. 457 (1939).

apply to dispersions in general is as follows:

$$\kappa = \frac{\log p/p_0}{\log h_0/h} \quad \text{............} (1)$$

where

κ = the coefficient of renitence,

p_0 = the yield value, or pressure required for incipient deformation, and

h_0 = the initial height of the cylindrical specimen.

Assuming that the volume of the specimen is constant under deformation, Eq. 1 is transformed to a more convenient form as follows:

$$\kappa + 1 = \frac{\log g/g_0}{\log h_0/h} \quad \text{............} (2)$$

where g = the load and p_0 is given by

$$p_0 = g_0 a_0 \quad \text{..........} (3)$$

where a_0 = the initial area of the specimen.

The coefficient of plasticity, π, is defined as the inverse of κ, as follows:

$$\pi = \frac{1}{\kappa} \quad \text{..............} (4)$$

The coefficient of plasticity, unlike yield value, is independent of the proportion of mixing water. The property of plasticity is defined numerically by the value of π, so that a dispersion with a higher value of π than another is the more plastic of the two, and *vice versa*.

Concretely considered, π is a principal determinant of the mechanical ease of plastic deformation. Plasticity is therefore genetically related to the same property, and a plastic dispersion is more easily deformed than one that is less plastic, and *vice versa*.

WORKABILITY OF MORTARS

Workability is a more complex property than plasticity and, in general, consists of several factors. Although the following discussion is directed specifically to mortars, it is to be understood that it applies substantially also to other disperse systems.

As the name implies, workability refers to the manner in which a mortar behaves, or "works," in practice. A prime factor of workability is plasticity, or, specifically, the pressure or work necessary to spread a mortar to a given thickness.

We shall now prove that π is the principal determinant of the plasticity factor for mortars (and other materials for which π is sufficiently small), and that p_0 may be neglected in this respect. The pressure or work of deformation is a function of both $1/\pi$ and p_0, but the dependence on $1/\pi$ is exponential and on p_0 algebraic, so that for π appreciably less than 1, it is the major variable. In addition to p_0 being the minor variable, its range of variation is moreover highly limited. This is readily understood when one considers the correlate of p_0, namely, the content of mixing water. The latter may be increased, and p_0 accordingly decreased, only to a narrow degree because of the limited water-carrying capacity of a mortar and because excess water is in general undesirable. Therefore, p_0 as both a minor and a limited variable is inconsequential as far as ease of deformation goes, and π when appreciably less than 1 is a proper and sufficient measure of the plasticity factor of workability.

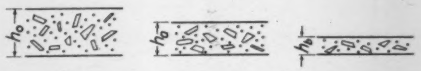

FIG. 1.—Stages in the Plastic Flow of a Mortar Which May or May Not Be Attainable.

A second factor of workability is the continuity of the deformation. After some extent of plastic flow, many mortars will rupture and break down rapidly into a narrow, hard cake. A required deformation may then be rapidly passed through without ever being attained. To illustrate the point, in Fig. 1 is represented a mass of mortar of height h_0, which it is desired to spread out to height h_a. However, as pressure is applied to the mortar, it ruptures at some thickness greater than h_a and breaks down rapidly to a smaller thickness h_b. Therefore this mortar is poorly workable from the standpoint of attaining the desired spread h_a.

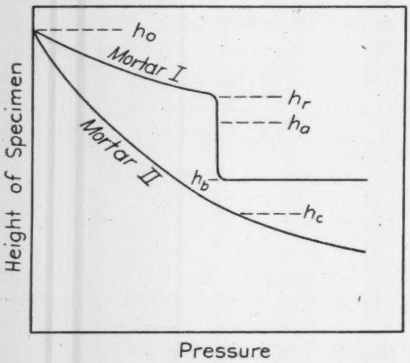

FIG. 2.—The Nonattainment of a Given Thickness During Plastic Flow Because of Rupture of the Mortar.

The point is brought out more definitely in Fig. 2, which is a representation of the graphic result obtained in the present method. For mortar I, rupture occurs at h_r, so that the specimen suddenly decreases in height from h_r to h_b, and it is impossible to realize intermediate height h_a as a stable configuration. On the other hand, for mortar II the deformation is continuous, and h_a is readily attained by application to the specimen of steady pressure.

A third factor involved in workability is the "hardening" of a mortar whereby plastic flow is suspended despite increase of pressure. Referring again to Fig. 1, starting with the mortar of height h_0, an increase of pressure causes it to spread out to a thickness, h_a, at which point suspension of flow occurs, so that the mortar seems to harden, and by virtually no increase in pressure can it be brought to a desired smaller thickness h_b.

Suspension of plastic flow is a common property of dispersions, and as regards mortars, is ordinarily due not to a premature setting of the cement but to the development (as in general) of a close-packed structure of the particles.

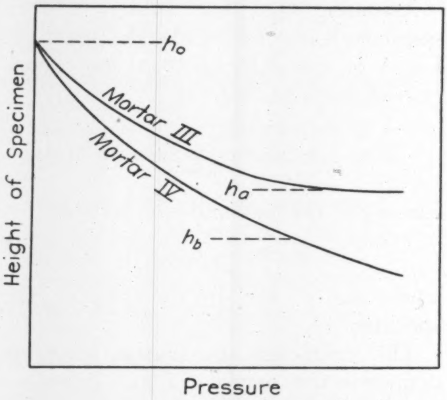

FIG. 3.—The Nonattainment of a Given Thickness During Plastic Flow Because of Suspension of Flow of the Mortar.

To illustrate the case more concretely, referring to Fig. 3, suspension of flow occurs for mortar III at height h_a, but for mortar IV the deformation by contrast continues beyond h_a as the pressure increases, so that thickness h_b, smaller than h_a, can be realized for this mortar, though not for mortar III.

A pseudosuspension of flow is that incidental to rupture, as shown for mortar I in Fig. 2. In this instance, the cake to which the mortar collapses after rupture is virtually maintained against increase in pressure, so that for all practical purposes the mortar has suffered a suspension of flow that prevents attainment of the smaller thickness h_c. Mortar II, on the other hand, can be deformed regularly to h_c.

A fourth factor of workability concerns the ability of a mortar to retain its mixing water under the suction of a brick. A mortar may be spread readily on a glass surface; but in contact with a brick, if its water retention is low, it may lose its mixing water too rapidly and will then be difficult to spread efficiently.

Of the four factors of workability, probably only a particular one of these will in any given instance be salient in determining the workability. One will say that the mortar is hard to spread, or that it is difficult to place due to breaking, or to a suspension of flow, or that it dries too rapidly on a brick. One may alter conditions so as to overcome one of these drawbacks, only to find perhaps that another appears. For example, one may find that, by adding more mixing water, the pressure required for spreading the mortar may be sufficiently reduced but that the mortar breaks in the range of thickness desired, or that it suspends before a required thickness is reached, or that on a porous brick it "sets up" too soon.

Now it is a significant fact that not only does π determine the work or pressure of spreading, but it also describes more or less the other three factors of workability. The theoretical reason for this is that all four factors are some function of the same physical variable, namely, the force of attraction between the grains of the mix,[3] and therefore all tend to vary in the same way. In other words, a mortar of low coefficient of plasticity will be hard to spread, it will rupture during plastic flow or will suspend its flow prematurely, and it will lose its mixing water readily. From this standpoint, the value of π for a mortar is not only a specific measure of its plasticity or the pressure factor of workability, but is, furthermore, a measure of its workability considered over all.

It will be understood, however, in view of the complex character of workability, that π can be only an approximate measure of this property, and that detailed consideration of workability will require a separate examination of each of the factors other than ease of spreading. Thus, when two mortars have the same value of π, they may still differ as to the tendency to rupture, or to suspend flow, or to retain water. Their workability differs, then, in a certain degree, which cannot be discriminated by π.

There is some question, however, whether such fine discrimination of workability, as enters when π is about equal for different mortars, is necessary in practice. When, however, the values of π for two mortars differ appreciably, then not only do the mortars differ as to ease of spreading, but they differ also in the same sense as far as the other factors go, and therefore in their overall workability.

When π is greater than about unity, the correlation of the various factors of workability then appears to be relatively poor, so that, for example, two materials that have the same value of π may differ very appreciably in their susceptibility to suspension of flow. Moreover, even the work or pressure of spreading requires for its consideration not only π, but also p_0, for example, in the simplest case, their ratio π/p_0. However, these considerations apply rather to materials like limes or plasters, and not to mortars, since for the latter π is invariably less than unity.

π and p_0 are obtained from a plot of log h against log g. Both breaking and suspension of flow are ascertainable from this plot, and also, especially the former, from the original experimental graph of h against g. Water retention is measured separately but apparently is satisfactorily indicated also as a function of p_0.

EXPERIMENTAL PROCEDURE

As the details of the apparatus[4] and method have been explained elsewhere,[3] only a general description will be given here, except for the special application to mortars. A cylindrical specimen of mortar is placed between two plates and subjected to a continuously increasing load, g. As the specimen is compressed, a record of its height, h, is obtained as ordinate on a sheet of coordinate paper fastened to a rotating drum. The abscissa of the paper is the load, g, which is calculable from the known rate of addition of lead shot and the known rate of rotation of the drum. The result of the experiment then consists in obtaining an h-g curve for the specimen under test.

In the present experiments, 28.3 g. of cement were incorporated with 91.7 g. of graded Ottawa sand, corresponding to a volume ratio of 1:3; water was added from a pipette, admixed with a spatula for about 1½ min., and the mass molded in a brass mold 34 mm. high (which represents the initial height of specimen h_0), 35 mm. wide at one end, and 37 mm. wide at the other. Probably a wider mold having a ratio of width to height of about 1.2:1 would have been more satisfactory. Depending on the uniformity of the specimen and of operating conditions, fairly large differences in the size and relative dimensions of the specimen resulted previously[3] in only slight or moderate changes in the value of π and p_0. The rate of addition of lead shot was 900 g. per min. Moderate changes in rate of loading were previously[3] found to affect π and p_0 to a small extent.

TYPES OF CURVE, AND DETERMINATION OF π AND p_0

Different types of mortars, and also different masonry mortars, yield characteristic h-g graphs. Change in the water content usually does not alter the characteristic shape of the curve.

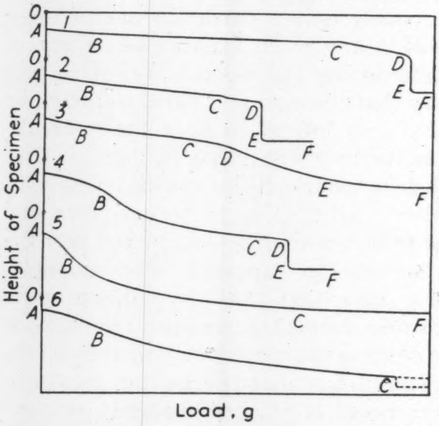

FIG. 4.—Types of Experimental h-g Curves for Mortars.

The more common forms of h-g curves for mortars are shown in Fig. 4. Curve 1 is typical of a 1:3 normal portland-cement mortar. Distance O-A represents the initial slump of the specimen due to its own superincumbent weight and to unavoidable squeezing during removal of the specimen from the mold. Section A-B is affected by these and other adventitious conditions and is unavailable for determining π and p_0; it is designated the "initial deviation," and usually extends to a pressure of about 10 g. per sq. cm. Section B-C, which is always concave and of varying length depending on conditions, represents plastic flow proper and is used to determine the magnitude of π and p_0. Section *C-D* represents a phase of prerupture; *D-E* represents actual rupture of the specimen and is called the "break." Section *E-F* comprises a range of pseudo-suspended flow.

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[4] A second model of the apparatus used in many of the present experiments differed from the original chiefly in having a counter balance for the load transmitter, to the latter being also attached the upper thin plate. The counterbalance improves the precision if the load transmitter is heavier than the load at the end of the "initial deviation." However, as regards the case of mortars, the original load transmitter was sufficiently light.

Curve 2 is characteristic of a 1:3 high-early-strength portland cement mortar and differs from curve 1 in that the curvature of section *B-C* is greater.

Curves 3 to 6 are characteristic of 1:3 masonry-cement mortars, 1:2 portland-cement mortars, and 1:3 portland-cement mortars with mineral-powder additions. Each of these types of 1:3 mortars may, however, exhibit a curve like that of curve 2, particularly when $\pi \times 100$ is less than about 10. Curve 3 is similar to curve 2, except that the break is more gradual. It has been found to pass completely over to curve 2 when the water content is decreased, π remaining, however, essentially the same. In curve 4, section *B-C* is highly concave and usually relatively short. Curve 5 is similar to 4 except that there is no apparent break, but suspension of flow may be present as indicated by section *C-F*. In curve 6, section *B-C* has less curvature than for either curve 4 or curve 5 and is longer, terminating either in a short break or in suspension of flow.

Curves 3 to 6, instead of being smooth, often are irregular or jerky, in which case the readings of h and g are not made at any arbitrary point but only just after an irregularity.[3]

A plot of log h against log g for determining π and p_0 is shown in Fig. 5. *A-B* corresponds to the initial deviation and *D-E* to the break; for a curve that shows no break, *D-F*, corresponding to suspension of flow, is obtained instead of *D-E*. Section *B-C* determines a characteristic straight line whose slope equals $-(\kappa + 1)$ and whose intersection with the horizontal, $\log h = \log h_0$, is $\log g_0$. Having found κ and g_0, π and p_0 are obtained by Eqs. 4 and 3.

Because of some confusion caused by presence of an initial deviation and of a break or suspension of flow, the establishment of line *B-C* in Fig. 5 must be made with caution so as not to include foreign end points located before *B* proper and beyond *C* proper. It is practically always necessary to have a series of curves for different water contents and to determine the respective *B-C* lines on the established principle that they are all parallel to one another and to some master line that represents the best average for all. With this precaution, an accuracy can be obtained that reflects the precision of operation and measurement. The error thus entailed in π and p_0, of the order of several per cent,[3] is quite small compared to the range in values for mortars.

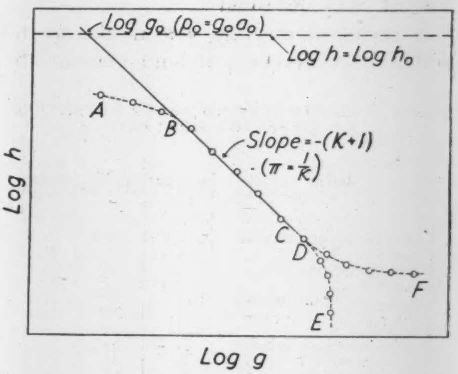

FIG. 5.—Log *h* - Log *g* Plot for Determining π and *p*.

COMPARISON OF PLASTICITY AND WATER RETENTION

It was desired to see how closely correlated are the plasticity and the water retention of mortars. The first problem was to measure the water retention. This was done by subjecting the mortar to suction, thereby simulating the action of a brick, and determining the loss of water from the mix. The specimen of mortar was placed in an 11 G 2 Jena glass filter and a suction of 2 in. mercury applied for 10 min., by which time the effect of further suction was relatively small. The filtrate was caught in a 10-ml. graduate placed in the suction flask and the volume noted. Water retention was defined as the milliliters of mixing water for which a filtrate of 5.4 ml. was obtained.

A series of mortars was made up in which part of the portland cement in a 1:3 mortar was replaced by an equal volume of powdered mineral; included in the series were a 1:2 portland cement mortar, a 1:3 masonry cement, and a 1:3 slag cement mortar. For the 1:2 mortar water retention could not be determined by the described method, owing to an anomalously small increase in filtrate with increase in mixing water.

TABLE I.—PLASTICITY AND WATER RETENTION OF DIFFERENT MORTARS.

Mortar	$\pi \times 100$	p_0 (16.7%)	Water Retention[b]
1:3 normal portland cement	2.6	0.9	21.0
½:½ talc:3	5.6	9.3	24.0
½:½ lime hydrate:3	6.7	16[a]	24.0
½:½ kaolin:3	8.3	20[a]	24.5
½:½ powdered diatomite:3	19	98[a]	27.0
¾:¼ ball clay:3	21	80[a]	26.5
⅞:⅛ bentonite:3	33	380[a]	27.0
1:2 normal portland cement	16	12	...
1:3 masonry cement	21	24	24.5
1:3 slag cement	21	13	24.5

[a] By extrapolation.
[b] Determinations by J. D. Neuss.

Results for $\pi \times 100$, p_0 at 16.7 per cent mixing water, and water retention are given in Table I. In Fig. 6 water retention is plotted against $\pi \times 100$. A fairly smooth curve can be drawn through the points for the various mortars with different mineral powder additions, indicating good correlation between plasticity and water retention for this group of materials.

The points for the masonry and slag cements fall definitely below the curve. This signifies that the water retention of these mortars is low relative to that of the mineral-powder mortars having the same plasticity.

Yield value of a mortar is a measure of the strength of the initial structure at the given water content. It is reasonable to suppose that the higher the water content at equal structural strength, the greater is the water retention, so that the content of mixing water at a given yield value may be taken as an index of the water retention. In practice, it is more convenient to employ instead of this the inverse quantity, yield value at a given water content. We have found that the reference water content is conveniently taken at 16.7 per cent of the dry weight of mortar; and the corresponding yield value, serving as an index of water retention, may then be written as p_0 (16.7 per cent).

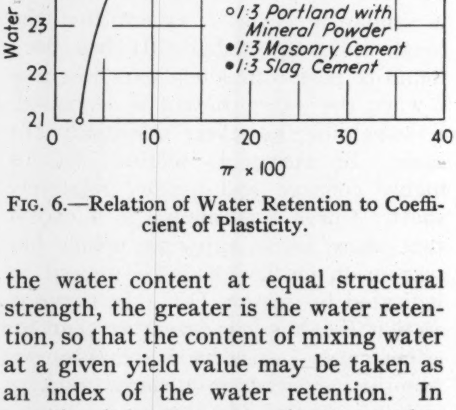

FIG. 6.—Relation of Water Retention to Coefficient of Plasticity.

By the definition of yield value, the deduction as to its relation to water retention applies strictly only at zero disturbance of the mortar. Because under practical conditions the disturbance is finite, π must be considered, and p_0 (16.7 per cent) as an index of water retention is subject to its magnitude. In other words, p_0 (16.7 per cent) may be compared for two mortars only when π is about the same for both. The greater p_0 (16.7 per cent) at equal π, the greater the water retention; if both p_0 (16.7 per cent) and π are greater, the indication is heightened, but if one is greater and the other less it is indeterminate.

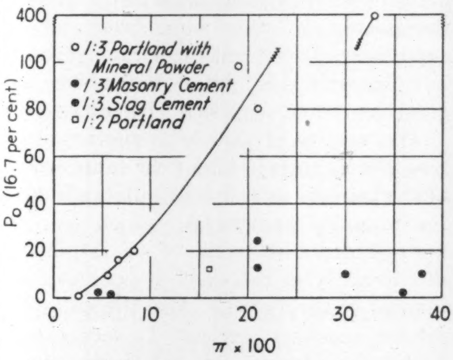

FIG. 7.—Relation of Yield Value at 16.7 per cent Mixing Water (as a Measure of Water Retention) to Coefficient of Plasticity.

From the data of Table I, p_0 (16.7 per cent) has been plotted in Fig. 7 against $\pi \times 100$. A smooth curve is obtained for the mortars with mineral-powder additions similar to that in Fig. 6 for water retention against $\pi \times 100$.

Included in Fig. 7 are points for the masonry cement mortars of Tables II and III, as well as for the masonry and slag cement mortars of Table I. The points for these fall below the curve for the mortars with mineral-powder additions, indicating again that their water retention is relatively low. A relatively low water retention seems to hold also for the 1:2 portland cement mortar (Fig. 7).

COMPARATIVE RESULTS FOR WORKABILITY

Although, owing to the complex character of workability, there can never be any single test that will perfectly characterize it, nevertheless, as was previously explained, coefficient of plasticity π may be viewed as a good approximation.

TABLE II.—COMPARISON OF MEASURES OF WORKABILITY, INCLUDING ALSO RESULTS FOR YIELD VALUE.

Mortar	$\pi \times 100$	p_0 (16.7%)	Penetration Method[a]	Bowl Method[a]
1:3 masonry cement......	42	2.3	18.9	102
1:3 high-early-strength cement..................	5.5	1.2	8.1	122
1:3 normal portland cement..................	1.9	0.1	6.1	135

[a] Reported by L. A. Wagner.

TABLE III.—COMPARISON OF MEASURES OF WORKABILITY OF DIFFERENT MASONRY CEMENTS, INCLUDING ALSO RESULTS FOR YIELD VALUE.

Mortar	Mason's Rating[a]	$\pi \times 100$	p_0 (16.7%)	Flow After Suction Method	Bowl Method[b]
1:3 masonry cement A...	Very good	38	10	65	105
B...	Excellent	36	2.1	81	108
C...	Excellent	30	10	90	106
D...	Poor	5.9	1.4	70	109
E...	Fair	4.1	2.2	52	107

[a] Reported by C. M. Butler.
[b] Reported by L. A. Wagner.

Measurements of π and p_0 were made on a group of masonry and portland cements. For purpose of comparison, results are included that were obtained by other tests of workability. In the Rogers flow-after-suction test,[5] the flow of a mortar is determined on the flow table, suction is applied to the mortar, its subsequent flow measured, and expressed as a percentage of the original.

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[5] Tentative Specifications for Masonry Cement (C 91 – 38 T), 1939 Book of A.S.T.M. Standards, Part II, p. 882.

In the Wagner bowl method,[6] water is added to the mortar in a bowl until a certain stirring resistance is attained, when the flow is measured on the flow table. In the Reed-Lewis penetration test,[7] the amount of water is measured at which a rod penetrates 50 mm. into the mortar.

Considering first a normal, high-early-strength, and masonry cement mortar, it is observed in Table II that π increases in the order named, indicating an increasing workability in this order, as expected. p_0 (16.7 per cent) also increases in the same order, indicating thereby, *a fortiori* in view of the concomitant increase in π, the expected order of increasing water retention.

The values obtained in the penetration and bowl tests also indicate that the workability increases in the sequence: normal, high-early-strength, and masonry cement mortar. The ratio of values for the extreme members is about 3 in the penetration test and only about 1.3 in the bowl test, whereas in the instance of π it is about 20, and therefore considerably greater. We believe that a very high ratio of values is consonant with the very marked difference in workability of these mortars. The main point is, however, that the higher the ratio the more sensitive is the test, and approximately in proportion to the ratio, assuming the precision of measurement to be about equal, as is reasonable in this case.

The workability of five different masonry mortars is compared in Table III. Included in the table are the ratings of an experienced mason. The mason's tests were made to obtain a reliable estimate of the relative behavior of these materials in practice and apparently were carried out with considerable care under comparable conditions. It is observed that three of the mortars for which π ranged between 30 and 38 were rated very good or excellent by the mason; on the other hand, two of the mortars for which π was only 5.9 and 4.1 were rated poor or fair. The values of π divide themselves virtually into two groups, and the mason's ratings seem likewise to divide themselves in the same way. The agreement of the mason's ratings and π is imperfect in detail but about as good as could properly be expected.

The results of flow-after-suction dispose the mortars in about the same order of workability as π, except outstandingly for masonry cement A, which by its unexplained low value of 65 is placed discordantly in the class of poor to fair instead of very good. Also the value of 70 for masonry cement D seems too high, considering its relatively poor workability.

The values obtained with the bowl plasticimeter do not appear to show any definite trend for these cements.

The values of flow-after-suction extend over about a twofold range, whereas those for π extend over about a nine-fold range. In view of the spread in mason's ratings from poor to excellent, the larger variation for π appears to be desirable and at any rate signifies a greater sensitivity of measurement.

No attempt is made to interpret the values of p_0 (16.7 per cent) in Table III because of the lack of a basis of comparison arising from the differences in the values of π, except to note that when p_0 (16.7 per cent) and π are both higher, the water retention may definitely be considered as higher.

The simple measure of workability furnished by π may be supplemented by further direct observations as to the factors of workability other than plasticity, employing the *h-g* curve, or a plot of log *h versus* log *g*, for this purpose. We shall here merely note the results for the masonry cements of Table III without further comment. Cement A showed a slight break or suspension of flow at the very end; the curve, which resembled curve 6 in Fig. 4, was somewhat irregular. Cement B exhibited suspension of flow and gave a smooth curve like that of curve 5. Cement C showed a decided break, and the curve, which was somewhat irregular, was like curve 4. Cements D and E both exhibited a sluggish break, as for curve 3, except that in the instance of cement E, there was at the lower water contents an abrupt break as for curve 2; the curves for cements D and E were both smooth.

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[6] Report of Committee C-12—Preliminary Consideration of Some Proposed Methods of Sampling and Testing Mortar for Unit Masonry, ASTM BULLETIN, No. 94, October, 1938, p. 40.

[7] E. W. Reed-Lewis, "Tentative Procedure for Determining the Relative Plasticity of Cements," *Pit and Quarry*, Vol. 33, No. 41, p. 48 (1941).

Although masonry cement C exhibited a break as in general for normal portland cement, the circumstances of the break differed characteristically from that for portland cement. In the instance of masonry cement C, the pressure at the break averaged about 700 g. per sq. cm., and the thickness was 55 per cent of its original value, falling to 35 per cent after the break. On the other hand, in the instance of the typical normal portland cement of Table II, the pressure averaged much higher at about 1800 g. per sq. cm., and the thickness at the break was only 90 per cent of its original value, falling to 60 per cent after the break. Therefore in the instance of the portland-cement mortar, the thickness cannot be reduced by steady pressure to any value between 90 per cent and 60 per cent of the original thickness, whereas in the instance of the masonry cement mortar it can be reduced uniformly to any value down to 55 per cent.

DISCUSSION

The reason for the apparent greater water retention, at the same plasticity, of the mortars with mineral powders as against the masonry mortars is to be connected with the inherent difference in constitution of the two systems. In the former, the plasticity is developed by the high surface area of the mineral powder, and in the latter, by the surface action of small amounts of organic admixtures. Each means is effective in promoting both plasticity and water retention. But the increase in water retention is relatively greater for the mineral powders, for the reason that the pore size is decreased by the increase in fineness, whereas no such effect is obtained with the organic admixture.

We may also view the question from the standpoint of the theory previously proposed of plastic flow of dispersions.[3] Both organic admixtures and finely divided solids increase the plasticity by virtue of the increase that they develop in the attraction between the dispersed particles in unit volume per unit distance of separation of the particles,—the organic admixtures by increasing the interfacial tension, and the powders by increasing the surface area. But, in the instance of the powders, not only is the attraction increased as a result of increase in surface area, but, in addition, the separation of the particles is decreased. A particular attraction is introduced thereby, which is uniquely effective in promoting yield value, or structural strength of the suspension, and accordingly water retention.

It is often considered that the action of organic admixtures in developing plasticity consists in their causing air to be whipped into the mortar during mixing. To test this point, measurements were made on a 1:3 mortar of slag cement, which in one instance was free of organic material and in the second instance was treated with an organic admixture. For the untreated cement, $\pi \times 100$ was determined to be 5.3, p_0 (16.7 per cent) to be 2.2, and the water retention to be 22.5 ml. For the treated cement, the respective figures were 21, 13, and 24.5. The organic admixture therefore markedly increased the plasticity. If the effect were one of causing air to be whipped into the mortar, one would reasonably expect the structural strength of the suspension to be less and therefore the yield value to be less. But actually p_0 (16.7 per cent) is much greater, which supports the explanation that the action of the organic admixture consists in causing an increase in attraction between the dispersed particles. For the same reaton, the directly measured water retension likewise is increased.

Acknowledgments:

The author is indebted to J. W. McBurney, National Bureau of Standards, for valuable discussions; to L. A. Wagner, Missouri Portland Cement Co., and C. M. Butler, Marquette Cement Manufacturing Co., for supplying data for comparison, and to J. D. Neuss, formerly of this station, for the determinations of water retention.

DISCUSSION

Mr. F. O. Anderegg.[1]—The definition of plasticity given in Mr. Roller's paper should be amended to include provision that the given shape shall be maintained after the deformation is completed.

The method described lacks the fundamental condition of constant stress application. With deformation occurring under varying stress, it is very difficult, if not impossible, to separate that effect from the effect of the time element.

Roller's yield point depends upon the conditions of the experiment. It will usually vary with the *rate* of stress application. For instance, a certain resin has a recovery of about 90 per cent when thrown on a solid floor, but can easily be deformed between the fingers by a slowly applied pressure. It is not a yield *point* but a yield *front* and the sooner this fact is realized the sooner can we begin to think straight.

It is respectfully suggested that the subject of the effect of organic admixtures on mortars is complicated by so many factors that conclusions should be drawn only with the greatest caution.

An important fundamental contribution to the subject of plasticity was presented to this Society twenty-one years ago, by Nutting.[2] It failed to receive any honor or recognition in his own country, but attention has been called to it recently in England,[3] where studies have been made on a variety of plastic substances. With the applied stress maintained constant, and with temperature under control, a systematic correlation has been obtained between the operator's psychological "feel" and the physical constants determined quantitatively in the laboratory. The concept of the "firmness" of the material, based on the Nutting equation permits an important tie-in between the human element or subjective and the physical objective. Anyone interested in the subject of plastics should study these contributions very carefully.

Mr. Paul S. Roller[4] (*author's closure*).—The "fundamental condition" postulated by Mr. Anderegg is applicable to materials that have a long time of relaxation. However, mortars deform like plastic solids having a very short relaxation time. Therefore their plastic behavior is properly described by a stress-strain relation, as in measurement of elasticity.

The resin cited as an example by Mr. Anderegg is a quite different material from a mortar, and deduction as to the latter may not be drawn from the behavior of the resin. When subjected to a small stress, at least in compression, a mortar will exhibit a definite strain whereas the resin apparently acts differently, like a supercooled liquid, and exhibits a continuously increasing strain. The yield value for a mortar does depend upon the rate of stress, but in a relatively small degree.

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[1] Consulting Specialist on Building Materials, Newark, Ohio.

[2] P. G. Nutting, "A Study of Elastic and Plastic Deformation," *Proceedings*, Am. Soc. Testing Mats., Vol. 21, p. 1162 (1921). Also "New Law of Deformation," *Journal*, Franklin Inst., Vol. 191, p. 629 (1921).

[3] G. W. Scott-Blair and F. M. V. Coppen, "The Measurement of the Firmness of Soft Materials," *Journal*, Soc. Chemical Industry, Vol. 60, p. 190 (1941); *Nature*, Vol. 149, p. 22 (1942).

[4] Physical Chemist, Bureau of Mines, U.S. Department of the Interior, Eastern Experiment Station, College Park, Md.

CURING OF MASONRY MORTARS

By Howard R. Staley[1]

Synopsis

This paper reports the effect on strength of 2-in. mortar cubes caused by variation in curing conditions. Fourteen variations in curing methods were used for lime-cement mortars and four were used for straight lime mortars. These variations in treatments include the effect of removal from the molds at 24 hr. and 48 hr.

A simple, economical storage has been devised using a converted ice refrigerator which yields results that are as consistent as obtained by using any of the other commonly used types of storage. The curing conditions obtained by using the converted ice refrigerator seem to be more suitable for all of the proportions of lime-cement mortars investigated than those obtained by any of the other storages used.

The storage most suitable for lime mortar specimens appears to be exposure to the laboratory air.

Since the lime-cement mortar proportions investigated are considered to represent the full range of characteristics that would be obtained in masonry mortars, there appears to be no need for water storage of masonry mortars at any time during the curing period.

Published data are rather meager regarding the effect on strength of masonry mortars produced by variations in curing conditions. The extreme range of values for strength reported by independent investigators, supposedly working with the same materials and proportions and subjecting the specimens to the same curing conditions, led the author to believe that variation in curing conditions was a contributing factor to the wide range of values. It has been the experience of the author that although a storage room may be strictly defined in a specification, practically any kind of room which is used for storage purposes may be considered to fulfill the requirements in some instances. It was the above factors, plus the attempt to find an economical and easily regulated storage that could be adopted by even the most poorly equipped laboratory, that prompted making the tests reported here.

Materials:

Three limes were used as follows:

No. 1—High-calcium hydrate.
No. 2—Autoclaved dolomitic hydrate.
No. 3—Regular dolomitic hydrate.

One brand of portland cement was used for all tests. Both the lime-hydrates and the portland cement were stored in airtight metal containers.

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[1] Assistant Professor of Building Construction, In Charge of Building Materials Research Laboratory, Massachusetts Institute of Technology, Cambridge, Mass.

Lime was considered to weight 45 lb. per cu. ft. and cement 94 lb. per cu. ft.

The sand was sieved to six sizes and recombined to an arbitrary gradation which follows closely the natural sands used for mortars in this area. The dry-and-loose weight of the sand was 95 lb. per cu. ft. The gradation used was as follows:

Sieve	Retained on Sieve, per cent
No. 8	3
No. 16	10
No. 30	25
No. 50	32
No. 100	24
pan	6

Proportioning and Mixing:

Mortars were mixed in the arbitrary proportions given in Tables IV and V. Proportions were selected to represent the full range commonly used for masonry mortars. For purposes of proportioning, the volumetric relations were transferred to weight relations, based on the weights given above.

The mortars were mixed by hand in a nonabsorbent bowl, the hands being covered with rubber gloves during mixing. Batch sizes varied from one using 1500 g. of sand to one using 4500 g. of sand. The following procedure was used in mixing:

1. Add water to the bowl.
2. Add the dry cementitious material to the water and mix with the fingers until thoroughly wetted.
3. Add sand to the wet slurry.
4. (*a*) 1500-g. batch; mix vigorously for 3 min.

   (*b*) 3000-g. batch; mix vigorously for 4 min.

   (*c*) 4500-g. batch; mix vigorously for 5 min.
5. Measure the flow. If the flow was not correct the batch was discarded and a new batch made.
6. Mold cubes.

Molding:

In molding the specimens, the procedure given in A.S.T.M. Tentative Method of Test for Compressive Strength of Portland-Cement Mortars (C 109 – 37 T)[2] was followed. It should be noted here that any commonly used method for filling molds results in a higher degree of packing of the high-cement mortars. It is impossible to obtain any packing action by these methods for the mortars of high lime content.

TABLE I.—PROCEDURE FOR CURING OF 2-IN. MORTAR CUBES.[a]

Type of Curing	Immediately After Molding	Time When Removed from Molds, hr.	After Removal from Molds	After Age of 7 Days
1	Left in laboratory air	24	To M.R.	In M.R.
2	Left in laboratory air	24	To water	In water
3	Left in laboratory, wet burlap cover	24	To M.R.	In M.R.
4	Left in laboratory, wet burlap cover	24	To water	In water
5	To M.R.	24	To M.R.	In M.R.
6	To M.R.	24	To water	In water
7	To M.R.	48	To M.R.	In M.R.
8	To D.R.	48	To M.R.	To D.R.
9	To M.R.	48	To water	In water
10	To D.R.	48	To D.R.	In D.R.
11	To D.R.	48	To D.R.	To water
12	To D.C.	48	To D.C.	In D.C.
13	To D.C.	48	To D.C.	To water
14	To D.R.	48	To D.R.	To D.C.
15	Left in laboratory air	48	In laboratory air	In laboratory air
16	Left in laboratory air	48	In laboratory air	To D.R.
17	Left in laboratory air	48	In laboratory air	To D.C.
18	Left in laboratory air	48	To D.R.	In D.R.

[a] M.R. = Moist room; 70 F., 95 per cent relative humidity (fog room).
D.R. = Damp room; 80 F., 80 per cent relative humidity.
D.C. = Damp closet; 70 F., 90 per cent relative humidity.
Water = In running water (in soapstone tank).
Laboratory air = Temperature and humidity variable.

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[2] 1939 Book of A.S.T.M. Standards, Part II, p. 892.

Curing of Cubes:

Treatment of the specimens after molding is given in Table I.

Sufficient water was always available in the moist room in the form of mist so that the specimens were continually wet on the surface.

In the damp closet and the damp room insufficient water was present to cause wetting of the surface of the specimens. A very economical damp closet can be constructed, as was done in this instance, from a second-hand ice refrigerator. This is shown in the photograph in Fig. 1. A burlap-covered frame was inserted vertically at the rear of the box and water fed onto this by means of a perforated pipe at the rear of the burlap. Shelves were constructed of hardware cloth in order that circulation would be impeded as little as possible. While much closer control was exercised over the conditions of temperature and relative humidity in the moist room and damp room, the variations of these factors in the damp closet were relatively small. Measurements made in the damp closet with a recording thermostat-humidistat showed a maximum variation of ± 8 F. and ± 5 per cent relative humidity.

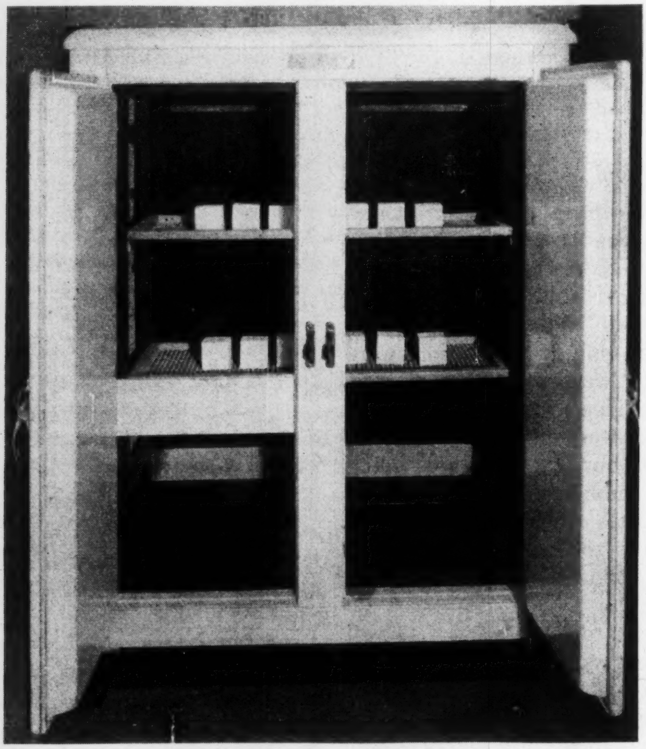

FIG. 1.—Damp Closet Storage.

Testing of Cubes:

Cubes were tested in a hydraulic testing machine.

Other Measurements:

The plasticity figures were obtained using the Emley plasticimeter with the procedure given in A.S.T.M. Tentative Method of Physical Testing of Quicklime and Hydrated Lime (C 110–38 T).[3]

Flow-after-suction was determined with the Rogers apparatus using the procedure given in A.S.T.M. Standard Specifications for Masonry Cement (C 91–40)[4], with the exception that the brick sand used for making the cubes was substituted for the Ottawa sand.

Sand-carrying capacity was measured using the extrusion device.[5] In the extrusion device a cylinder of mortar is extruded under pressure through a truncated conical section. Those proportions that can be extruded without excessive pressure are termed workable.

Fineness of the limes was determined using the wash test given in A.S.T.M. Tentative Method C 110 – 38 T.[3]

RESULTS

The characteristics of the cementitious materials are given in Table II. Lime No. 2, the autoclaved dolomitic hydrate, shows the highest plasticity figure and sand-carrying capacity. It is generally true that a lime with a high plasticity figure has a high sand-carrying capacity. However, the apparent relationship existing between plasticity figure and sand-carrying capacity for the three limes used is not generally true for all limes. This latter statement is based upon results already published[6] and upon measurements on a large number of limes, the results of which are as yet unpublished. Flow-after-suction is high for the three limes and based upon the measurements mentioned above this is characteristic of all limes, while that for the portland cement is relatively low as would be expected. A large percentage of lime No. 2 is retained on the No. 200 sieve in the wash test and while the percentage retained is still within the limits specified by A.S.T.M. standards, it is higher than the usual percentage for hydrates. A very light brushing of the sieve while the washing is being carried out decreases this to approximately 5 per cent, indicating that the known agglomerating or flocculating characteristics of this particular lime exists even with a large amount of water present. There is no doubt but that some of the characteristics of this lime are due entirely to the tendency of the lime particles to flocculate in the paste.

TABLE II.—CHARACTERISTICS OF CEMENTITIOUS MATERIALS.

	Emley Plasticity	Flow After Suction,[c] per cent	S.C.C., by weight[a]	Fineness, per cent retained on	
				Sieve No. 30	Sieve No. 200
Portland cement......	...	59	1C:2.28S	...	7.50[b]
Lime No. 1.........	156	91	1L:7.38S	0.00	5.04
Lime No. 2.........	340	96	1L:12.00S	0.34	14.44
Lime No. 3.........	125	95	1L:5.80S	0.10	3.77

[a] Sand-carrying capacity (based upon dry materials).
[b] Mill test.
[c] Flow after suction test made with the combined masonry sand used for mortars in accordance with the procedure given in A.S.T.M. Standard Method of Test for Water Retention of Masonry Mortar (C 91 – 40)

Table III gives the average values for several measurements on the mortars used. The requirements for mixing water to produce the desired consistency varied in approximately the same manner for the three limes, the requirements increasing in each instance with increasing lime content. The density of the mortars decreased with increasing lime

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[3] 1939 Book of A.S.T.M. Standards, Part II, p. 910.

[4] 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 1.

[5] Walter C. Voss, "Lime Characteristics and Their Effect on Construction," Symposium on Lime, p. 103, Columbus Regional Meeting, Am. Soc. Testing Mats. (1939).

[6] H. R. Staley, "Properties of Limes and Lime Mortars," *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 975 (1941).

TABLE III.—DATA ON MORTARS.

Mix, by volume C:L:S	Lime No. 1			Lime No. 2			Lime No. 3		
	Water, per cent[a]	Flow, per cent	Density, lb. per cu. ft.[b]	Water, per cent[a]	Flow, per cent	Density, lb. per cu. ft.[b]	Water, per cent[a]	Flow, per cent	Density, lb. per cu. ft.[b]
1:0.25:3	14.72	111	135.0	14.75	109	133.0	15.18	116	132.0
1:1:5	15.82	111	131.8	16.68	110	124.0	16.27	112	125.0
1:1.5:6.25	17.29	113	130.0	18.29	111	123.0	17.97	115	124.0
1:2:7.50	17.50	113	129.2	17.95	108	122.0	17.41	111	123.0
1:3:10	18.36	114	128.7	19.77	108	121.0	19.04	112	123.0
0:1:2.50	25.00	118	120.5	24.70	101	113.0	26.20	115	113.7

[a] Mixing water; based on weight of dry material in batch.
[b] Density of freshly mixed mortar.

content as would be expected, but with the mortars containing limes Nos. 2 and 3 the decrease in density was much greater than for those mortars made with lime No. 1. These data indicate that there is more entrapped air in the mortars made with limes Nos. 2 and 3. Calculations based on these data indicate that there is 1.5 to 2.0 per cent more entrapped air in the mortars containing limes Nos. 2 and 3 than in that containing lime No. 1 at the proportion of 1:0.25:3. At the proportion 1:3:10 there appears to be about 5 per cent more entrapped air in the mortars containing limes Nos. 2 and 3. For any one type of lime, cement, and sand, with the same method for curing, there is a straight-line relation between strength of cubes and the density of the freshly-mixed lime-cement mortars. This is shown in Fig. 2 where density is plotted against 28-day strength. These data indicate that for a given set of materials and conditions, it would be possible to predict the strength with some degree of accuracy merely by measuring the density of the freshly mixed mortars. If this relationship is generally true then it would only be necessary to measure the strength and density of two or three mixes and the strength of any other proportion could be found by measuring the density and interpolation at any point along the line. With different sand, cement, and lime than those used in these tests, the variation in strength would be somewhat different.

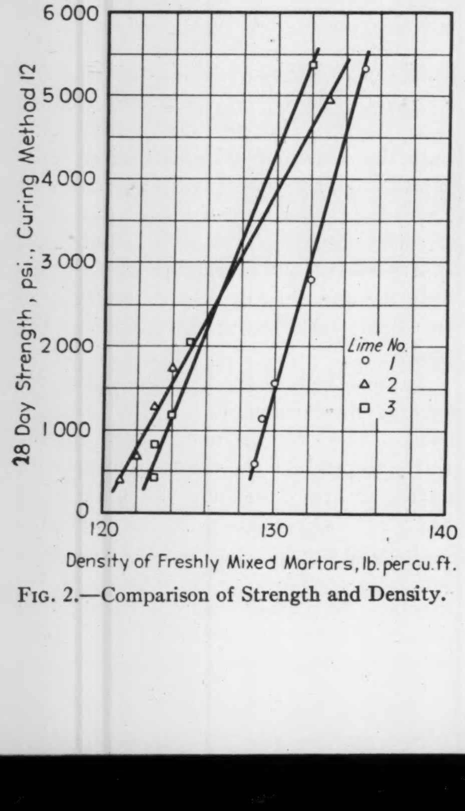

FIG. 2.—Comparison of Strength and Density.

The results of the compression tests of the 2-in. cubes are given in Tables IV and V where each value given for any one lime represents the average value for 3 cubes. Wherever results appeared questionable, the mixes were repeated to make certain that some error had not been made in preparation or curing of the specimens.

The data given in Table IV are pre-

TABLE IV.—COMPRESSION TESTS, 2-IN. CUBES.
Each result given is average of 3 cubes

Mix, by volume C:L:S	Age, days	Lime	Compressive Strength, psi.													
Type of curing			1	2	3	4	5	6	7	8	9	10	11	12	13	14
1:0.25:3	7	No. 1	3557	3127	3306	2831	3316	2885	4358	3377	2932	2669	2668	4061	4200	3277
		No. 2	3225	2794	2772	2574	2565	2485	3732	3072	2435	2310	2316	3399	3560	3086
		No. 3	3066	2826	2725	2623	2679	2602	3693	3027	3294	2762	2760	3895	4116	3397
		Avg.	3280	2915	2935	2675	2855	2655	3925	3160	2885	2580	2580	3785	3955	3255
	28	No. 1	4163	4481	3820	4136	3831	4118	6119	5260	4091	3528	3454	5311	5425	3855
		No. 2	4185	4087	4308	3802	4030	3467	5042	4688	3632	2428	3293	4967	4851	3016
		No. 3	4273	3890	4024	3940	4011	3761	5343	4977	4495	4034	2760	5356	4826	3144
		Avg.	4205	4155	4050	3960	3955	3950	5500	4975	4070	3330	3200	5210	5035	3335
1:1:5	7	No. 1	1256	1171	1044	1066	1417	1568	1437	1328	1037	1674	1592	2023	2066	1628
		No. 2	1391	1181	957	920	1049	861	909	899	973	1109	1138	1237	1239	1257
		No. 3	1444	1285	1066	1021	1319	993	1244	962	1307	1159	1243	1517	1521	1349
		Avg.	1365	1210	1020	1000	1260	1140	1195	1065	1105	1315	1325	1590	1610	1410
	28	No. 1	2946	1887	1904	1673	2364	2157	2158	2341	1708	1850	2350	2793	2546	1957
		No. 2	2029	1735	1651	1388	1603	1260	1330	1688	1521	1204	1485	1771	1575	1240
		No. 3	1903	2076	1590	1387	1840	1363	1732	2208	1495	1304	1709	2023	1810	1290
		Avg.	2290	1900	1710	1480	1935	1595	1740	2080	1575	1450	1845	2195	1975	1495
1:1.5:6.25	7	No. 1	876	470	606	340	530	349	818	751	727	982	926	910	937	1080
		No. 2	467	489	354	346	327	323	710	568	351	650	723	845	841	731
		No. 3	535	557	402	385	359	342	683	583	455	740	724	742	742	668
		Avg.	625	505	455	355	405	340	735	635	510	790	790	830	840	825
	28	No. 1	1460	880	1114	658	1081	671	1406	1646	1160	1076	1306	1539	1399	1104
		No. 2	775	855	630	622	637	586	1189	1249	509	888	950	1280	1092	805
		No. 3	897	1018	783	714	723	653	1010	1221	715	810	918	1184	1009	667
		Avg.	1045	915	840	665	815	635	1200	1370	795	925	1050	1335	1165	860
1:2:7.5	7	No. 1	722	...	516	...	459	...	481	634	418	649	663	657	651	653
		No. 2	620	...	445	...	432	...	403	409	313	489	508	449	448	460
		No. 3	633	...	432	...	391	...	427	505	333	645	580	527	544	590
		Avg.	660	...	465	...	425	...	435	515	355	610	585	545	545	565
	28	No. 1	1200	...	829	...	785	...	783	1236	686	787	859	1139	912	905
		No. 2	972	...	728	...	680	...	583	907	457	544	650	698	586	529
		No. 3	809	...	601	...	528	...	604	1117	493	701	673	813	663	669
		Avg.	995	...	720	...	665	...	655	1085	545	675	725	885	720	700
1:3:10	7	No. 1	149	...	80	...	78	...	171	237	311	516	467	329	342	475
		No. 2	150	...	93	...	86	...	108	170	149	297	285	237	239	248
		No. 3	182	...	115	...	98	...	99	188	156	225	220	265	280	343
		Avg.	160	...	95	...	85	...	125	195	205	345	325	275	285	355
	28	No. 1	272	...	183	...	145	...	318	675	467	627	581	582	385	432
		No. 2	256	...	160	...	160	...	190	407	218	316	330	342	264	237
		No. 3	337	...	221	...	193	...	189	499	246	257	318	417	332	417
		Avg.	290	...	190	...	165	...	230	525	310	400	410	445	325	360

TABLE V.—COMPRESSION TESTS—2-IN. CUBES, LIME MORTARS (1 LIME, 2.5 SAND).
(Each result given in average of 3 cubes)

Age, days	Lime	Compressive Strength, psi.			
Type of curing		15	16	17	18
7	No. 1	105	103	100	62
	No. 2	96	98	93	62
	No. 3	88	72	74	49
	Avg.	95	90	90	55
28	No. 1	153	122	72	110
	No. 2	176	119	68	120
	No. 3	115	89	47	94
	Avg.	150	110	60	110

sented graphically in Fig. 3. Considering the 1:0.25:3 mix, it is obvious that during methods 1 to 6, inclusive, and 9 to 11, inclusive, do not produce optimum strengths for this type of mortar. The treatments given in methods 7, 12, and 13 yield approximately the same results and it appears that any one of these methods would be suitable for this type of mortar. The consistently low values for treatments 1 to 6 may be due to removing the molds at 24 hr. The only direct comparison of the effect of removing the molds at 24 hr. is given by curing treatments 5 and 7, and 6 and 9. The difference is very marked between methods 5 and 7 and the same tendency is indicated between 6 and 9 though the difference is much less. It is quite evident that the low humidity afforded by treatments 10, 11, and 14 is not conducive to optimum strength for this proportion. The average percentage increase in strength from 7 to 28 days is approximately the same for all treatments except 10, 11, and 14.

that an early drying period might result in higher values than those obtained by some of the other methods. Here again the low humidity of treatments 10, 11, and 14 again is not conducive to optimum strength, although it is indicated that the water storage after 7 days for treatment 11 offsets somewhat the early exposure to low humidity.

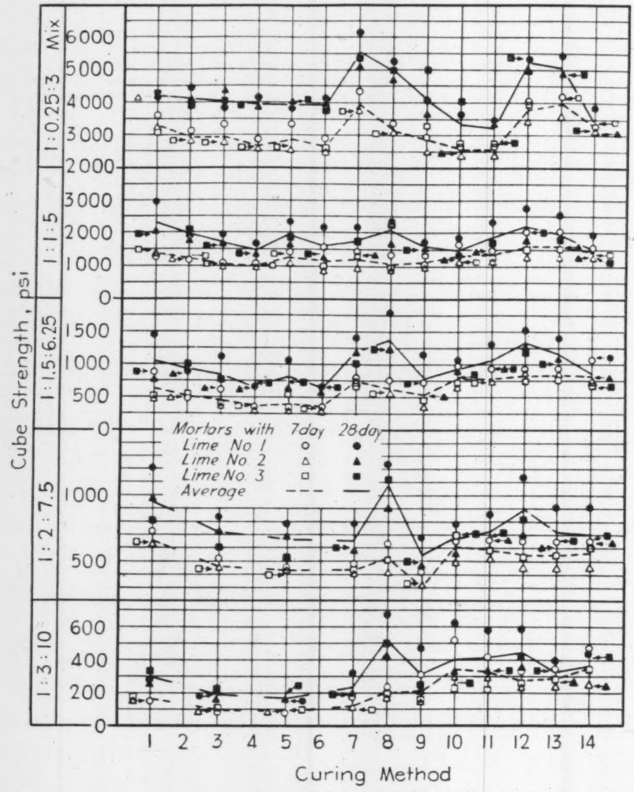

FIG. 3.—Comparison of Strength of Cubes with Variation in Curing Method.

For the 1:1:5 mix the same trend is hown, although not quite so markeds, as shown for the 1:0.25:3 mix. The effect of removing the molds at 24 hr. is evidently lessened somewhat by the drying effect of exposure to the laboratory air for this period under methods 1 and 2. The difference in strength produced by removing the molds at 24 or 48 hr. appears to be negligible in this instance, although it is indicated

The 1:1.5:6.25 mix reacts in approximately the same way to the varied treatments as the 1:1:5 mix, except that the effect of the drier atmosphere in the latter part of the curing period of treatment 8 results in a higher percentage increase in strength from 7 to 28 days. The decrease in strength due to immersion in water is somewhat more marked than for the previous mix.

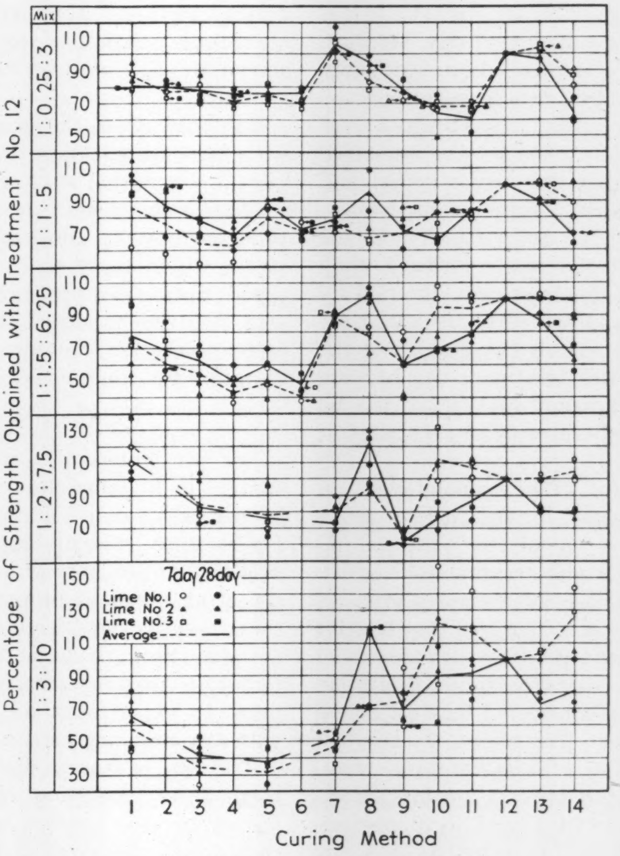

FIG. 4.—Comparison of Curing Methods.

For the 1:2:7.5 mix, the effect of removing the molds at 24 hr. is noticeably reduced by the drying period afforded by treatment 1. No tests were projected for treatments 2, 4, and 6 for this or the 1:3:10 mix as it was assumed that still lower results would be obtained than given for the other treatments. Treatment 8 resulted in the highest 28-day strength, indicating again that the lower humidity in the latter half of the curing period was conducive to increase in strength although the 7-day strength obtained with this treatment is lower than for some of the other treatments. The lower 7-day strength is no doubt due to the exposure to the wet atmosphere of the moist room. Early immersion in water under treatment 9 gave the lowest results, and the cubes which were immersed in water at any time had a chalky surface at the time of removal. Storage of this mix in water is not conducive to optimum results as shown under treatments 9, 11, and 13.

The effect on strength of removal from the molds at 24 hr. is quite obvious for the 1:3:10 mix and again some benefit is obtained by the early drying period afforded under treatment 1. Treatment 8 results in the highest strength although the 7-day strength is lower than obtained with some of the other treatments. It is rather surprising that treatment 9 gave higher strength values than 7 and equivalent 7-day strength to method 8. These results are probably questionable, but insufficient time was available to repeat these mixes. All cubes of this mix which were immersed in water during the curing period had chalky surfaces at times of removal.

Considering all proportions and the application of any single curing method to all mixes, the treatment afforded by method 12 appears to be as uniformly suited as any. It is the opinion of the author that this treatment can be very easily and economically duplicated by any other laboratory for comparative purposes.

Accordingly, the percentages of strength obtained for the various curing methods were calculated with the results obtained with treatment 12 as 100 per cent. These values are plotted in Fig. 4. Here it can be seen that relative to the strengths obtained with treatment 12, there is no other single treatment that could be considered to yield equivalent results. Treatment 1 for the 1:1:5 and the 1:2:7.5 mortars, treatment 8 for the 1:2:7.5 mortars, treatments 10 and 11 for the 1:3:10 mortars, treatments 7 and 13 for the 1:0.25:3, and treatment 13 for the 1:1:5 mortar might be said to yield results equivalent to those obtained with treatment 12. The lower humidity afforded by treatments 10 and 11 affect the relative strength percentages the least for the mortars of higher lime content, while the water storage after 7 days of treatment 13, or the moist curing of treatment 7, affects the relative percentages least for the mortars of higher cement content. Considering the effect of all the curing methods the largest relative percentage variation of strength is shown by the mortars of higher lime content.

Table V gives the results obtained with the straight lime mortars. It is indicated here that storage in the laboratory air is the correct treatment for this type of mortar. Since the laboratory air to which these specimens were exposed averaged approximately 30 per cent relative humidity, storage in air of relatively low humidity seems to be desirable for this mortar.

CONCLUSIONS

For the lime-cement mortars tested, storage in a damp closet at 70 F. and 90 per cent relative humidity, with no fog or spray present, appears to yield results that are normal and consistent. Since the range of proportions used in these tests is considered representative of the full range of characteristics usually found in masonry mortars, there appears to be no need for water storage with any masonry mortar.

The type of storage devised for curing method 12 consisting of a converted ice refrigerator appears to yield results that are as consistent as those obtained by any of the other types of storage used.

The curing treatment best suited for straight-lime mortars appears to be storage in laboratory air with a fairly low relative humidity.

DISCUSSION

MR. F. O. ANDEREGG.[1]—Some of the data presented by Mr. Staley afford an opportunity for making a plea for uniformity in conventions for mortar proportions. His "1:1:5" mix is made up with 94 lb. cement, 45 lb. of hydrated lime and 475 lb. of sand. According to the convention adopted last year by Committee C-7 on Lime,[2] such a mix would be 1:0.9:5.93 by volume, while according to a recent proposal from Committee C-12 on Mortars for Unit Masonry this same mix would become 1:1$\frac{1}{8}$:5.93. Should not those of us in this Society who are interested in mortars get together and agree to talk the same language?

I should like to ask Mr. Staley, in view of his practical experience as a brick mason, to state whether all mortars made up to a given flow would be suitable for laying bricks which have (*a*) low, (*b*) medium or (*c*) high initial absorption rates. Our experience has been that, according to the amount of sand and lime in the mix, and depending upon the initial suction of the bricks, flows ranging from about 75 to as high as 140 per cent would be suitable for use. Some data presented recently by Fishburn[3] have a bearing on this point.

MR. J. W. McBURNEY[4] (*by letter*).—Has the author any data which indicate the effect of water content at time of testing on apparent compressive strength? What, for example, would be the effect of adding a 24- or 48-hr. air drying period following removal from water at 28 days?

Has Mr. Staley any data on the water retentivities of the mortars themselves? Although the author states that flows after suction reported in Table II were made in accordance with the procedure given in A.S.T.M. Standard Specifications for Masonry Cement (C 91 – 40),[5] the values more resemble "flows after suction" than "per cent of initial flow" as prescribed by Specifications C 91 – 40. The unusually high values for the limes may be explained by the richness of the mortars (45:135 by weight, or approximately 1:1$\frac{1}{2}$, by volume). Even so, the values are notably higher than those reported by Wells.[6] Wells tested lime mortar 1:6 by weight (approximately 1:3 by volume) and presented results as "flow after suction" where the initial flow was 130 per cent. Differences in proportioning do not, however, explain the very high value given for portland cement. Compare the 59 per cent water retentivity reported by Mr. Staley on a straight portland cement mortar with the 31 per cent (G2) and 34 per cent (H2) values reported for 1:1:6 mortars in Table IV of Committee C-12's cooperative report.[7] These low water retentivities are checked by other laboratories. Some cooperative checking of instruments and methods would seem advisable. From the data available, water retentivities reported by Mr. Staley appear to be about 20 per cent above those usually reported. Interestingly enough, the Emley plasticimeter reading appears to be in the opposite direction. This statement is based upon lime No. 2 of the present paper and data presented by Mr. Staley before the Society in 1941.[8]

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[1] Consulting Specialist on Building Materials, Newark, Ohio.

[2] Report of Committee C-7 on Lime, *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 277 (1941).

[3] Cyrus C. Fishburn, "Water Permeability of Walls Built of Masonry Units," *Report BMS 82*, Building Materials and Structures, April 15, 1942.

[4] Senior Technologist, National Bureau of Standards, Washington, D. C.

[5] 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 1.

[6] Lansing S. Wells, Dana L. Bishop and David Watstein, "Differences in Limes as Reflected in Certain Properties of Masonry Mortars," *Journal of Research*, Nat. Bureau Standards, Vol. 17, No. 6, December, 1936, p. 895 (*RP952*).

[7] See p. 266.

The compressive strengths are in good agreement with the C-12 data where water contents of the mortars agree. Consideration might be given to the possibility that, if Mr. Staley and the writer should trade their testing instruments, it might be found thereafter that they had also traded their views as to what are reasonable requirements for water retentivity and strength of particular mortar mixes.

Permit me to say, in conclusion, that Mr. Staley's paper is very timely, informative and valuable.

MR. HOWARD R. STALEY[9] (*author's closure*).—In answer to Mr. Anderegg, I agree that there should be some uniform practice adopted in regard to mortar proportions. My personal preference would be a definition on a weight basis, using the weights of dry materials in the proportion rather than a volumetric basis where different interpretations can be used.

Mortars of all proportions of cement-lime content, when made up to the same flow, would not be equally suitable for laying brick in any one of the classifications given in (*a*), (*b*), or (*c*). Considering any one classification, it is generally true that mortars of high cement content require a higher flow as measured by the flow table to be suitable for the mason's requirements. The variation in flow or consistency between a high-cement and a high-lime mortar would be larger when used with the brick of high initial absorption due to the fact that water retentivity is a more critical factor in this range of absorption. That is, the high-cement mortar would have a tendency to become rigid more quickly, due to loss of water, than the high-lime mortar, therefore more water must be added. In the case of any one mortar the flow usually must be greater the higher the rate of absorption of the unit being laid. In all probability the range of flow values would be at least as high as indicated by Mr. Anderegg.

There is a general lack of appreciation of the fact that there is a very small degree of precision in the average mason's judgment regarding flow or consistency, for the reason that the mason becomes accustomed to using any one mortar over a fairly wide range of consistency. When the mortar is first placed on the board it is probably slightly wetter than would be considered "normal" consistency. By the time the mortar has been used up, the consistency is stiffer than would be considered "normal." The flow range might well be as high as 40 or 50 per cent during the period of using the mortar. Unless the mason has had considerable practice in mixing a mortar from dry materials and adding water in small increments until a workable consistency is obtained, his judgment regarding consistency is apt to be valueless from a laboratory control standpoint.

Replying to McBurney, no data are available for the exact conditions indicated by him but some additional work has been done which includes the drying effect for longer periods. The effect of laboratory storing (curing method 15) has been extended to include all the proportions investigated and two additional methods have been used as shown under curing methods 19 and 20 below:

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[8] H. R. Staley, "Properties of Limes and Lime Mortars," *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 975 (1941).

[9] Assistant Professor of Building Construction, In Charge of Building Materials Research Laboratory, Massachusetts Institute of Technology, Cambridge, Mass.

Type of Curing	Immediately After Molding	Time When Removed from Molds, hr.	After Removal from Molds	After Age of 7 days
19...	Left in laboratory	48	To D. C.	In D. C.
20...	Left in laboratory	48	To D. C.	In laboratory air

The results obtained for the cement-lime mortars using the three additional methods are given in the accompanying Table I.

From this Table, it can be seen that the curing conditions afforded by exposure to laboratory air the entire time (method 15) does not give maximum results for any of the cement-lime mortars and is very detrimental to the strengths of the mortars of high cement content. Curing methods 19 and 20 also result in a decrease of strength, below that obtained with curing method 12, for the 1:0.25:3 and the 1:1:5 mortars. For the 1:1.5:6.25 mortars and curing methods 19 and 20, the 7-day strengths are low, while the 28-day strengths are about 80 per cent of those obtained with curing method 12. For the 1:2:7.5 and 1:3:10 mortars, the use of curing methods 19 and 20 results in low 7-day strengths while the 28-day strengths average lower for curing method 19 and somewhat higher for curing method 20 than obtained with curing method 12.

TABLE I.—COMPRESSION TESTS.

Mix by Volume C:L:S:	Age, days	Lime	Compressive Strength, psi.		
Type of Curing.................			15	19	20
1:0.25:3	7	No. 1	2734	2980	2978
		No. 2	2200	2452	2474
		No. 3	2403	2654	2758
	Avg...........		2445	2695	2735
	28	No. 1	2656	3872	4024
		No. 2	2150	3398	3386
		No. 3	2248	3579	3580
	Avg...........		2350	3615	3660
1:1:5	7	No. 1	1200	1364	1367
		No. 2	859	992	988
		No. 3	890	969	1009
	Avg...........		980	1105	1155
	28	No. 1	1171	2077	2230
		No. 2	890	1513	1577
		No. 3	1012	1570	1665
	Avg...........		1025	1720	1825
1:1.5:6.25	7	No. 1	750	818	819
		No. 2	522	572	570
		No. 3	535	550	582
	Avg...........		600	645	655
	28	No. 1	765	1285	1355
		No. 2	592	899	962
		No. 3	586	950	1012
	Avg...........		645	1045	1110
1:2:7.50	7	No. 1	476	505	516
		No. 2	397	382	397
		No. 3	405	382	395
	Avg...........		425	420	435
	28	No. 1	472	895	906
		No. 2	448	676	729
		No. 3	465	716	794
	Avg...........		460	760	895
1:3:10	7	No. 1	318	298	314
		No. 2	208	179	175
		No. 3	226	210	215
	Avg...........		245	225	230
	28	No. 1	359	529	625
		No. 2	242	317	370
		No. 3	287	379	505
	Avg...........		295	405	500

It is practically certain that a 24 or 48-hr. drying period just before testing would increase the strength of the specimens over that obtained with wet specimens but it is believed that such a procedure would not give strengths with the full range of mortars comparable to those obtained by using curing method 12.

No data are available on the water retentivities of the mortars themselves. The flow after suction values given are "per cent of initial flow" as specified in A.S.T.M. Specifications C 91 – 40. The richness of the mortars used may account for the values obtained as compared to those obtained on mortars by others. I have suggested to Committee C-12 that considerable cooperative work could be done with the Rogers apparatus to determine the degree of correlation obtained by various laboratories and the manner in which the flow after suction value is altered by various procedures. It should be remembered that the flow after suction value of 59 was obtained using the standard proportion according to the

Federal Specification for Cement; Masonry (SS - C - 181b)[10] and A.S.T.M. Specification C 91 - 40, rather than on the mortars as reported to Committee C-12. No one knows the effect of proportion of sand, richness of mix or brand of cement or of lime on these results, and variation in these materials would certainly affect the results obtained. Lime No. 2 of the present paper was not included in the results reported in 1941, though the statements made then would apply equally as well to this lime. The same tendency regarding the Emley Plasticimeter reading is shown now as was indicated in 1941.

I heartily agree that there should be some means of checking laboratory instruments and procedures. All of the instruments used by this laboratory are supposedly standard instruments built and assembled by reputable manufacturers, with the exception of the flow table which was assembled in our own shop. We have obtained excellent reproducibility in our results, therefore the laboratory techniques involved cannot be a major factor in this laboratory. The Cooperative Tests on Masonry Mortars reported by Committee C-12[11] give some idea as to the wide variation that is obtained in results by laboratories working with supposedly standard instruments and it is the belief of the author that no specification for masonry mortars can be intelligently formulated until more work of this kind is completed. The proper place to start such work appears to be in the calibration of instruments used in the various laboratories, rather than in the manner previously used.

Contrary to Mr. McBurney's statement, I have made no recommendations regarding reasonable requirements for water retentivity or strength of various proportions of mortar in this paper. On the basis of these results, I have some definite opinions on this subject but I have confined the subject matter of the paper to reporting the results as obtained.

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[10] Published by U. S. Government Printing Office, January 12, 1938.

[11] See p. 266.

FACTORS INFLUENCING THE REFLECTIVITY OF WHITE PORTLAND CEMENT CONCRETES

By C. W. Muhlenbruch[1] and E. H. Miller[1]

Synopsis

The reflectivity of white cement concretes now in wide use for curbs and other reflecting surfaces is affected by types of aggregates, water-cement ratio, method of curing, and method of finishing. Freezing and thawing, and exposure tests of 150 summer and 100 winter days show that loss of reflectivity may be as high as 70 per cent. Various ratios of light and dark sand to cement give information useful in designing a mix. The effect of five dispersing or wetting agents on reflectivity, normal consistency, and strength has been studied. Wetting agents when added in the amount of 1 per cent by weight of mixing water overcome the difficulty of mixing white waterproof type cement without affecting the properties.

White concrete has been used extensively for curbing and center line markers in highway work to provide contrast with ordinary gray concrete. It has also been used for terrazzo floors, architectural concrete, and more recently for light-reflecting floors in airplane assembly plants. For "saw-tooth" reflecting curbs of some types the concrete is cast against a form and for floors or center line markers it is trowled to the final finish. In most cases maximum reflectivity is desirable even under severe conditions of exposure. While considerable attention has been given to the method of installing white concrete, little has been published regarding the factors which contribute to its reflectivity or "whiteness."

The primary purpose of this investigation was to determine the influence of the water-cement ratio, quantities and types of aggregates, and severe conditions of exposure on the reflectivity of white portland-cement concrete. A secondary study was made of the effect on reflectivity, normal consistency, and strength caused by the addition of various admixtures intended to increase the workability of the mixes and thereby to increase their reflectivity.

Materials and Test Specimens

Two types of white cement commonly available were studied: namely, standard white and waterproof white. The waterproof type is ground with calcium stearate to cause a reduction in capillary movement of water through the set concrete. The two cements used had the following general properties:

	Standard White	Waterproof White
Normal consistency, per cent	25.6	30.0
Vicat setting time, hr.		
Initial	1.92	1.50
Final	4.67	4.25
Tensile strength, 7 days, psi.	400	376
Compressive strength, 7 days, 2-in. cubes, psi.	2950	3100
Autoclave expansion, per cent	0.03	0.04
Specific surface, sq. cm. per g.	2130	1910

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[1] Assistant Professor of Civil Engineering, and Senior in Civil Engineering, respectively, Carnegie Institute of Technology, Pittsburgh, Pa.

Allegheny River sand, which has a light brown color, and commercial white sand ground to a natural grain from a 99½ per cent silica rock deposit near Mapleton, Pa., were the two types of fine aggregate studied. The following sieve analyses, made in conformity with A.S.T.M. Standard Method of Test for Sieve Analysis of Fine and Coarse Aggregates (C 136 – 39),[2] were obtained for the fine aggregate:

Sieve	Percentage Passing	
	White Sand	River Sand
No. 4	100	99
No. 8	100	98
No. 16	100	90
No. 30	97	81
No. 50	17	17
No. 100	2	1

The only coarse aggregate used was washed crushed limestone having the following sieve analysis made in conformity with A.S.T.M. Method C 136:

Sieve Size	Percentage Passing
1½ in.	100
1 in.	80
¾ in.	31
½ in.	15
⅜ in.	6
No. 4	2

In addition to the sieve analyses the following general properties were determined for all aggregates in accordance with the appropriate A.S.T.M. procedures:

	White Sand	River Sand	Limestone
Unit weight, lb, per cu. ft.[a]	94	98	95
Percentage of voids[b]	43	37	43
Apparent specific gravity[c]	2.66	2.49	2.67
Fineness modulus[d]	1.84	2.13	...

[a] Standard Method of Test for Unit Weight of Aggregate (C 29 – 39), 1939 Book of A.S.T.M. Standards, Part II, p. 308.

[b] Standard Method of Test for Voids in Aggregate for Concrete (C 30 – 37), *Ibid.*, p. 310.

[c] Standard Method of Test for Specific Gravity and Absorption of Coarse Aggregate (C 127 – 39), *Ibid.*, p. 300. Standard Method of Test for Specific Gravity and Absorption of Fine Aggregate (C 128 – 39), *Ibid.*, p. 302.

[d] Standard Specifications for Concrete Aggregates (C 33 – 40), 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 87.

A total of 224 reflectivity specimens 8 in. square and 2 in. thick were cast in lightly paraffined cardboard boxes. The mix was composed of one part by volume of white portland cement, two parts by volume of fine aggregate, and three parts by volume of coarse aggregate with water-cement ratios of 5, 6, 7, and 8 gal. per sack. The quantity of fresh concrete required to cast the four test blocks prepared for each water-cement ratio constituted a batch. The weights of material required to yield one batch were calculated by the absolute-volume method to permit actual proportioning by weighing. Specimens having a water-cement ratio of 5 gal. per sack were placed with a vibrator and all exposed surfaces were finished by troweling. Two of the four specimens were cured for 28 days in a moist room maintained at 100 per cent humidity and 70 F., while two were cured under room conditions for the same length of time. Specimens cured in the humidity box were permitted to dry two days before measuring the reflectivity.

Because of the natural repellency to water of the waterproof white cement, this material was added after the water had been mixed with the sand. The coarse aggregate was then added to the mix. While it is possible to mix the waterproof cement with water, it requires considerably more time than the method used and was therefore considered impractical for actual work.

Five admixtures were used with both types of cement in an effort to increase the reflectivity, particularly for the lower water-cement ratios, by bringing the cement particles to the surface as a result of increased workability of the concrete mix. The admixtures were dispersing agents, wetting agents, grinding aids, or penetrants as follows:

Admixture Type 1.—A dispersing agent containing triethanolamine and about 98 per cent soluble in water at 130 F. Ground with cement or dissolved in mixing water in ratio of 4 oz. per barrel of cement.

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[2] 1939 Book of A.S.T.M. Standards, Part II, p. 298.

Admixture Type 2.—A natural resin about 90 per cent soluble in water at 130 F. Occasionally used as a grinding aid. Dissolved in mixing water in ratio of 3 oz. per barrel of cement.

Admixture Type 3.—A penetrant used to hasten the dyeing of fabrics. Completely water soluble at ordinary temperatures. Dissolved in mixing water in varying concentrations.

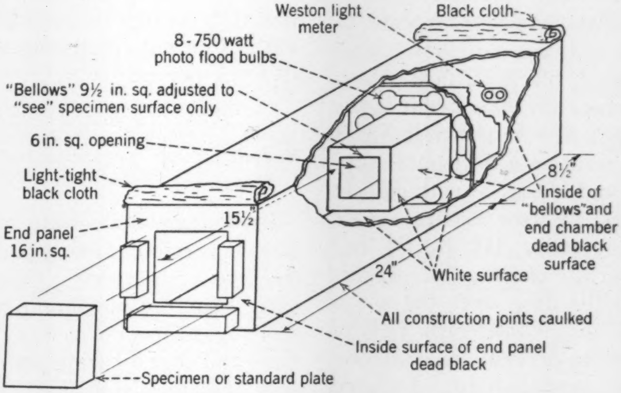
Weston light meter
Black cloth
8 - 750 watt photo flood bulbs
"Bellows" 9½ in. sq. adjusted to "see" specimen surface only
6 in. sq. opening
Light-tight black cloth
End panel 16 in. sq.
15½"
8½"
Inside of "bellows" and end chamber dead black surface
24"
White surface
All construction joints caulked
Inside surface of end panel dead black
Specimen or standard plate

FIG. 1.—Apparatus Used to Determine the Reflectivity of White Portland Cement Concretes.

Admixture Type 4.—A synthetic wax used to promote a spreading effect in casein paints, shoe dressings, etc. Completely water soluble at 135 F. Ground with cement or dissolved in mixing water in ratio of 4 oz. per barrel of cement.

Admixture Type 5.—A wetting agent used in textile dyeing and for photographic purposes. Completely water soluble at ordinary temperatures. Obtained as a 10 per cent solution and dissolved in the mixing water.

APPARATUS

The tests for normal consistency, time of set, tensile strength, compressive strength of 2-in. cubes, autoclave expansion, and fineness or specific surface were made in conformity with A.S.T.M. Standard Methods of Sampling and Physical Testing of Portland Cement (C 77 – 40),[3] Tentative Method of Test for Compressive Strength of Portland Cement Mortars (C 109 – 37 T),[4] Tentative Method of Test for Autoclave Expansion of Portland Cement (C 151 – 40 T),[5] and Tentative Method of Test for Fineness of Portland Cement by Means of the Turbidimeter (C 115 – 41 T).[6] In making the normal consistency test for the waterproof cement, it was necessary to increase the mixing time to about 6 min. to permit enough combination of water and cement to give uniform results.

Grinding of the admixtures with the cement was accomplished in porcelain jars charged with flint pebbles and 5 lb. of cement. The grinding was continued for 30 min. at a speed of 35 rpm.

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[3] 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 27.

[4] 1939 Book of A.S.T.M. Standards, Part II, p. 892.

[5] 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 176.

[6] 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 257.

The apparatus used to measure the reflectivity of the white concrete specimen is shown in Fig. 1. The "bellows" is adjusted by moving it toward or away from the specimen until only the light from the test surface is reflected through the 6-in. square opening. This adjustment need only be made once for the size of specimen used. The inside top, bottom, and two side surfaces of the specimen end of the box are painted white to diffuse the light completely over the test surface. The inside surface of the specimen end of the light box, the inside of the "bellows," and the inside of the back chamber have been exposed to the smoke of burning camphor. This produces a dead black surface which absorbs approximately 99 per cent of the light falling upon it. As shown in Fig. 1 the specimen rests on a horizontal supporting block and is aligned with respect to the opening by two vertical guide blocks. This opening ($7\frac{3}{4}$ by $7\frac{3}{4}$ in.) gives a test surface of 60 sq. in. Light-tight black cloths drop over the specimen and over the photoelectric cells of the light meter to prevent the measurement of any outside light. Diffused light from the eight 750-w. Photoflood bulbs falls upon the test surface and is reflected through the "bellows" to the two Weston photometer cells where it is measured by a Weston illumination meter reading to 0.1 foot candle.

All readings are referred to a standard plate made of sand-blasted white glass and having a reflectivity of 82 per cent which value was obtained with a reflectometer. The procedure is to place the standard plate across the specimen end of the light box and obtain a reading for the amount of reflected light. A test specimen then replaces the standard plate and the reflected light is again measured. The ratio of this latter value to the standard value, when multiplied by 0.82, is the actual reflectivity of the specimen.

When a surface that has been smoked black with burning camphor is placed in the test position at the specimen end of the box the reflected light is less than $\frac{1}{2}$ foot candle. This indicates that the "bellows" is properly placed and that the photometer cells measure only the light reflected from the test surface. The values of reflectivity obtained with this apparatus are within 3 per cent of those obtained with a reflectometer. Values of reflectivity differing by only a few per cent may be considered the same inasmuch as light-measuring equipment will not permit comparisons better than about 3 per cent.

RESULTS AND DISCUSSION

Reflectivity of Neat Cement and Cement Mortars:

Preliminary tests were made on neat cement specimens using plain water and the five admixtures. This study indicated that the maximum reflectivity of room-cured specimens was reached in 15 days and that a curing period of 28 days was therefore sufficient. The average reflectivity for these neat cement specimens was 82 per cent with no one type varying from the average by more than 2 per cent. These values may be compared with 33 per cent for gray portland cement mixed with white sand and 20 per cent for the same cement using river sand.

Effect of Admixtures on Physical Properties of Cements:

Pilot specimens prepared in outlining the tests indicated that the 5 gal. per sack mix would be difficult to work even with a vibrator. The five admixtures previously mentioned were used in an attempt to increase the workability of this mix. It was felt that an admixture which, when added to the mixing water, would reduce the normal consistency of a cement would tend to produce increased workability if the same amount of mixing water was used. The effect of four of these admixtures upon the normal consistency of standard white cement is shown in Fig. 2. Admixture type 3 was the most effective in reducing the normal consistency.

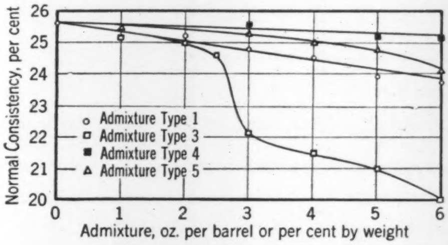

FIG. 2.—Effect of Various Admixtures upon the Normal Consistency of Standard White Cement.

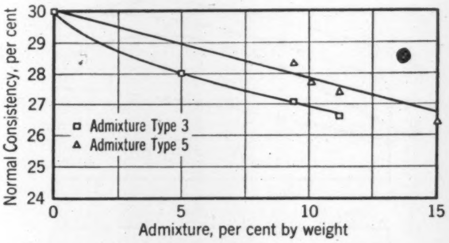

FIG. 3.—Effect of Penetrant and Wetting Agent upon the Normal Consistency of Waterproof White Cement.

All the admixtures except type 3 had practically no effect upon the 7-day tensile and compressive strengths. Type 3 showed no change up to 3 per cent concentration but at this point the strength began to decrease until at 6 per cent concentration the tensile strength was 20 per cent lower and the compressive strength of 2-in. cubes was 30 per cent lower. Because of this reduction in strength admixture type 3 was not used with the standard type of white cement.

The penetrant, which is admixture type 3, and the wetting agent, which is admixture type 5, were used in an attempt to reduce the normal consistency of the waterproof cement with the results shown in Fig. 3. Their effect upon the 7-day strength of waterproof cement is shown in Fig. 4. Although the compressive strength was raised and the tensile strength was lowered, the added workability and ease of mixing resulting from the use of these admixtures was the primary consideration. Both admixtures, although they satisfied the conditions for normal consistency at any concentration, actually seemed to produce excess workability so that the cement particles were brought to the surfaces during molding, leaving a relatively weak core. Since 1 or 2 per cent of the penetrant or wetting agent was enough to overcome the mixing difficulty of the waterproof type of cement, the reflectivity specimens were made with these concentrations.

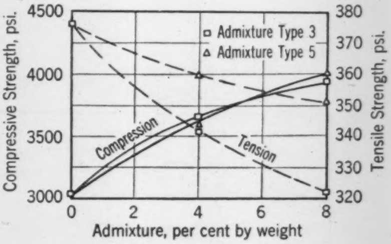

FIG. 4.—Effect of Penetrant and Wetting Agent upon the 7-day Strength of 2-in. Cubes and Briquets Made with Waterproof White Cement.

Effect of Aggregate on Reflectivity:

Clean, sound coarse aggregate has no effect upon the reflectivity of white concrete if it is not worked to the surface. The sand used does have an effect, however, as may be seen from Fig. 5. As the sand content of the mortar is increased to a ratio of one part cement, four parts sand, the reflectivity of about

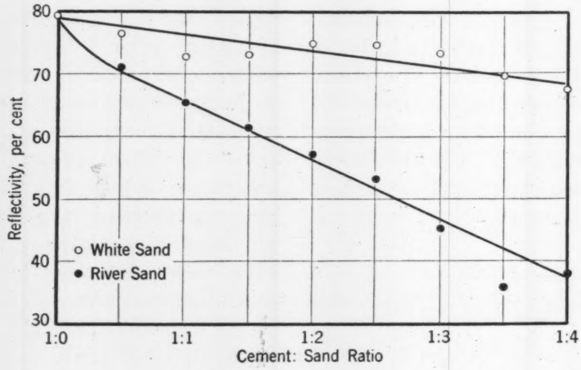

FIG. 5.—Effect of Various Amounts of White and Allegheny River Sand on the Reflectivity of White Cement Mortars.

Each plotted point represents the average of two specimens.

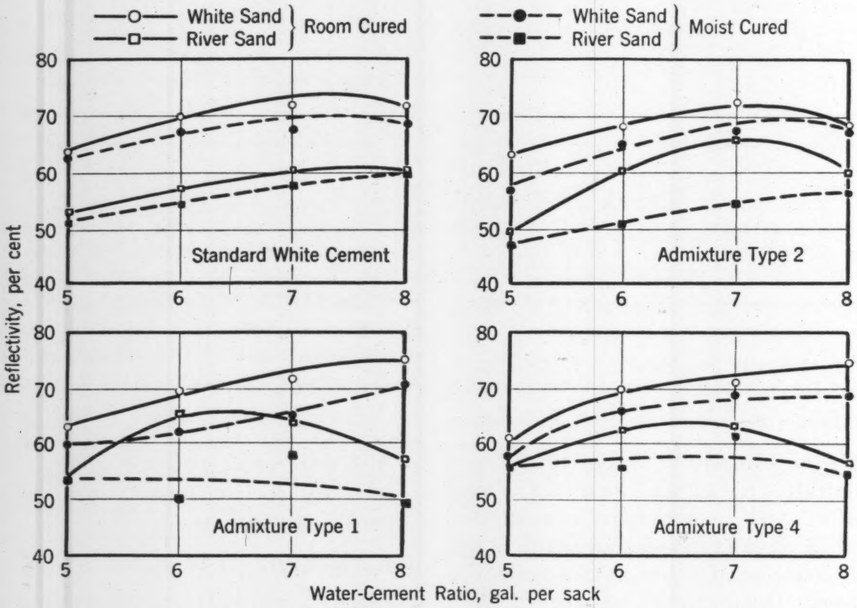

FIG. 6.—Characteristic Reflectivity Curves for Increasing Water-Cement Ratios. Admixtures added to mixing water. Trowled surfaces only.

Each plotted point represents the average of two specimens.

80 per cent decreases to about 38 per cent for river sand and to about 68 per cent for white sand. Figure 5 shows that a 1:0.5 river sand mortar has the same reflectivity as a 1:3.3 white sand mortar. For white mortar the choice of a sand will depend, as it also does for the white concrete, on the relative costs of the two and the yield produced using the proper cement-sand ratio.

Effect of Water-Cement Ratio, Method of Curing and Finishing, and Admixtures on Reflectivity:

In Fig. 6 are shown characteristic curves for the relationship between reflectivity of troweled surfaces and water-cement ratio for standard white cement using river sand or white sand in moist-curing or room-curing conditions and three types of admixtures. The admixtures were dissolved in the mixing water at the temperatures previously indicated. When admixtures types 1 and 4 were ground with the cement the same reflectivity values were obtained for the white concrete as was obtained when the admixtures were dissolved in the mixing water. For the 7 and 8 gal. per sack water-cement ratios the trowled surfaces were glazed and shiny. That part of the test specimen which was cast against the mold was from 8 to 35 per cent lower in reflectivity than the trowled surface, averaging about 20 per cent lower. The finishing operation with the trowel not only evens up or smooths out minor roughnesses of texture but also causes cement particles to rise, producing both a smoother, or more reflective, and a whiter surface.

In most cases the results for reflectivity of duplicate specimens were in close agreement for the trowled surfaces, seldom differing by more than 5 per cent. The surfaces cast against the mold, however, were not so uniform, occasionally differing by 15 per cent.

The amount of mixing water is the most important factor, aside from the quantity and type of sand, which influences the reflectivity. For maximum whiteness enough mixing water is necessary to permit the cement particles to be brought to the surface by working and to secure a smooth, even, trowled surface. That the admixtures have not improved the workability enough to give increased reflectivity for the white sand concretes may be seen from Fig. 6. Also they have not affected the 5 gal. per sack river sand mixes, but have slightly increased the reflectivity of the intermediate water-cement ratio groups for this type of sand. The reflectivity of the trowled surfaces of room-cured specimens with a water-cement ratio of 5 gal. per sack and admixture type 5 averaged 65 per cent, approximately the same as the specimens mixed with plain water.

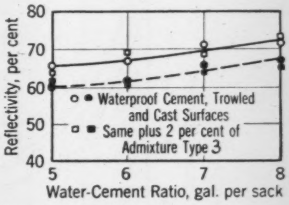

FIG. 7.—Reflectivity Curves for Waterproof White Cement Using White Sand.

Each plotted point represents the average of two specimens.

As may be seen from Fig. 6, moist-cured specimens were from 5 to 15 per cent darker than those cured under room conditions. White sand produces a block from 5 to 20 per cent higher in reflectivity than river sand, averaging about 15 per cent "whiter." From Fig. 5 the white and river sand for the 1:2 ratio used in these specimens differ in reflectivity by about 17 per cent which agrees with this average difference and shows that the coarse aggregate has little effect upon reflectivity. A worn or ground surface would undoubtedly show a greater difference since the river sand particles or coarse aggregate would actually be exposed.

Figure 7 gives reflectivity values for room-cured waterproof white cement and white sand specimens having water-cement ratios of 5, 6, 7, and 8 gal. per sack. These specimens are of approximately the same whiteness as those obtained with the plain white cement. It will be noticed, however, that the trowled and "as cast" surfaces are almost the same, which was not the condition encountered with the plain white cement specimens. The 2 per cent of admixture type 3 did not increase the reflectivity, but it did permit the waterproof cement to combine readily with the mixing water. Concretes using this type of cement may be mixed in the usual way if the admixture is added to the mixing water. Without the admixture, however, it is necessary to mix the water with the sand and then add the cement and coarse aggregate as mentioned previously. This latter method has the further disadvantage of requiring considerable time for complete mixing.

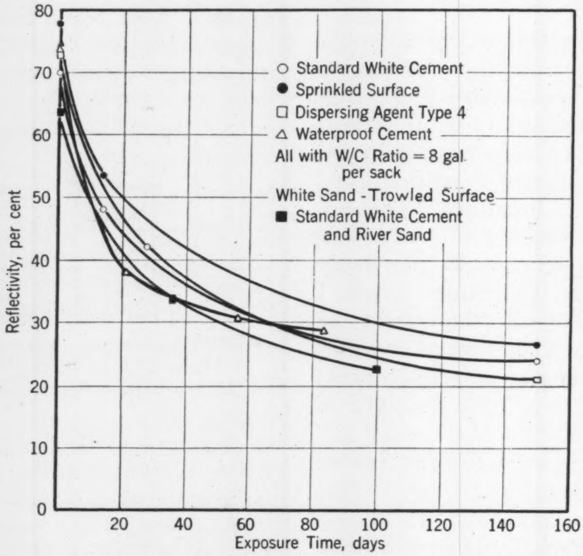

FIG. 8.—Loss in Reflectivity for Various Periods of Atmospheric Exposure.

Waterproof cement and standard white cement with river sand were exposed in winter. Other specimens exposed in summer.

A few specimens were prepared from ordinary gray portland cement, river sand, with a water-cement ratio of 8 gal. per sack. Just before final set took place, the surface was sprinkled with standard white cement. The quantity applied in this manner was equivalent to 1 lb. per sq. yd. The surface reflectivity of the specimens prepared in this way was 78 per cent. This method would be satisfactory only for architectural purposes and then only under mild exposure conditions, as will be pointed out.

Effect of Wetting on Reflectivity:

All of the white concretes tested showed loss in reflectivity due to wetting.

Specimens were placed in clean tap water at room temperature for 10 min. and then removed until they were surface dry. Some representative values, before and after wetting, for 8 gal. per sack mixes follow:

	Reflectivity, per cent Before Wetting	Reflectivity, per cent After Wetting
Standard white cement and white sand	72	56
Same plus admixture type 1	75	51
Same plus admixture type 4	74	54
Waterproof cement and white sand	71	61
Same plus 2 per cent of admixture type 5	70	59

TABLE I.—WHITE CONCRETE SUBJECTED TO ATMOSPHERIC EXPOSURE.

Water-cement ratio of 8 gal. per sack with white sand. Trowled surfaces. Each value represents the average of two specimens.

Specimen type	Exposure Period, days[a]	Reflectivity, per cent			Loss in Reflectivity, per cent[b]	Gain in Reflectivity by Cleaning per cent[b]
		Original	Final	After Cleaning		
Standard white cement	150 S	72	24	24	67	0
Same plus admixture type 1	150 S	75	21	31	72	13
Same plus admixture type 1	100 W	75	20	34	73	19
Same plus admixture type 4	150 S	74	22	40	70	24
Same but using river sand (no admixture)	100 W	62	22	22	65	0
Same plus admixture type 4 and river sand	100 W	74	17	20	77	4
Standard waterproof cement	85 W	71	31	36	56	7
Same plus 2 per cent of admixture type 3	85 W	72	35	38	51	4
Sprinkled surface	150 S	78	27	35	65	10

[a] S indicates summer and W indicates winter exposure.
[b] Expressed as a percentage of the original reflectivity.

The waterproof cement with or without an admixture is affected the least by wetting. The concentration of admixture used has not reduced the repellent action of the calcium stearate.

Effect of Atmospheric Exposure on Reflectivity:

The effect of atmospheric exposure upon reflectivity for 150 summer days and 100 winter days is shown in Fig. 8. These and other test results are summarized in Table I. Limits for reflectivity, as shown by Fig. 8, indicate that, when subjected to atmospheric exposure, the standard white cement using river sand gives the "darkest" concrete, while the waterproof cement with white sand is somewhat whiter. The specimen with the sprinkled surface was badly surface-crazed after 150 summer days of exposure. When the specimens were cleaned with a mild soap powder the reflectivity was increased in most cases, as may be seen from Table I. The admixtures were of little benefit in reducing the loss in reflectivity but seemed to give a surface that responded better to cleaning. The waterproof cement, with or without admixtures, showed the least reduction during exposure but did not respond to surface cleaning so well as the standard white cement with some of the admixtures. It appears that all types of white concrete subjected to atmospheric exposure might reduce to the same reflectivity over long periods of time.

Effect of Freezing and Thawing on Reflectivity:

White concrete specimens were subjected to two types of freezing and thawing. In a mild form this consisted in subjecting the specimens to a temperature of 30 F. for 12 hr. and then raising this temperature to about 40 F. for 12 hr. A more severe condition was obtained by subjecting the blocks to a temperature of 20 F. for 12 hr. and then spraying them with tap water at 120 F. until they were surface warm.

A representative summary of the results of freezing-and-thawing tests is included in Table II. The loss in reflectivity caused by mild freezing and thawing was about the same for all specimens and averaged 31 per cent. Severe conditions produced a greater loss as can be seen from Table II. Admixture type 3 was very effective in reducing this loss. The losses in reflectivity for the "as-cast" surfaces are not given in Table II but were approximately the same as the trowled surfaces for all mixes except those with a water-cement ratio of 8 gal. per sack. Moist-cured specimens decreased in reflectivity about the same as those cured under room conditions. The mix with the 8 gal. per sack water-cement ratio lost its glazed trowled surface when subjected to severe freezing and thawing which accounts for a greater loss in reflectivity than in the case of the "as-cast" surfaces. At the conclusion of the tests the trowled and cast surfaces were about the same for the 8 gal. per sack mixes.

TABLE II.—WHITE CONCRETE SUBJECTED TO CYCLES OF FREEZING AND THAWING.
Specimens made with white sand. Trowled surfaces. Each value represents the average of two specimens.

Specimen type	Water-Cement Ratio, gal. per sack	Cycles of Freezing and Thawing[a]	Reflectivity, per cent		Loss in Reflectivity, per cent[b]
			Original	Final	
Standard white cement	6	150 M	70	48	31
	8	150 M	72	47	35
Same plus admixture type 1	6	150 M	69	47	32
	8	150 M	75	50	33
Same plus admixture type 4	6	150 M	70	50	29
	8	150 M	74	53	28
Standard white cement	5	35 S	64	38	41
	7	35 S	72	52	28
Same plus 2 per cent of admixture type 3	5	35 S	64	52	19
	7	35 S	68	54	21
Standard waterproof cement	5	35 S	66	45	32
	7	35 S	71	54	24

[a] M indicates mild and S indicates severe freezing and thawing.
[b] Expressed as a percentage of the original reflectivity.

All of the specimens were sound except those made of standard white cement with a water-cement ratio of 5 gal. per sack. These specimens were disintegrating badly on the surface after 35 cycles of severe freezing and thawing. This mix was very difficult to place, even with a vibrator, and had a porous surface that readily absorbed water. Freezing after sprinkling the surface with water, therefore, was most effective for these specimens. Because of the tighter original surface the specimens with a water-cement ratio of 7 gal. per sack were not so badly disintegrated on the surface as the 5 gal. per sack specimens and did not decrease so much in reflectivity. The waterproof white cement has been reduced in reflectivity almost as much as the standard white.

SUMMARY AND CONCLUSIONS

The data presented in this paper seem to justify the following conclusions for standard white cement and waterproof white cement:

1. The maximum reflectivity of neat white cement and of white concrete is reached in 15 days and is not appreciably affected by admixtures in the form of dispersing agents, grinding aids, penetrants, or wetting agents whether added to the mixing water or ground with the cement. The admixtures may be helpful, however, in increasing the workability of the low water-cement ratio concrete mixes and increasing the strength of standard tensile briquets or 2-in. compression cubes. The reflectivity of neat white cement is 82 per cent and the average for concrete mixes made with white sand is 72 per cent.

2. As the water-cement ratio is increased, the reflectivity of white cement concretes is increased, reaching a maximum between 7 and 8 gal. per sack. This is due to the better workability of these mixes which permits the particles of white cement to rise to the surface and which also produces a smoother surface.

3. Clean, sound coarse aggregate has no effect on the reflectivity of white concrete while fine aggregate does. Mortar specimens made with four parts of white sand will decrease the "whiteness" 12 percentage points and river sand will decrease it 42 percentage points for the same proportions. White concrete having a 1:2:3 mix by volume when made with river sand will be an average of 17 percentage points lower in reflectivity than the same mix made with white sand.

4. White concrete cast against a paraffined mold will average 20 percentage points lower in reflectivity than a trowel-finished surface, indicating that the faces of saw-tooth light-reflecting curbs should be finished with a forming tool rather than cast to shape.

5. Moist-cured white concrete will be from 5 to 15 percentage points lower in reflectivity than room-cured specimens.

6. Wetting white concrete will decrease its reflectivity an average of 19 percentage points. Concrete made with waterproof type cement is not affected in this regard so much as the standard type.

7. When subjected to atmospheric exposure, white concrete may lose as much as 77 per cent of its original reflectivity. Subsequent cleaning with a mild soap solution may raise the reflectivity as much as 24 per cent or it may not be effective in recovering any of the original "whiteness." It appears that all types of the white concrete tested might reduce to the same reflectivity after long periods of atmospheric exposure.

8. Severe cycles of freezing and thawing may reduce the reflectivity of white concrete as much as 41 per cent.

9. Wetting agents added in the amount of 1 or 2 per cent by weight will overcome the difficulty of mixing waterproof type white cement without affecting the properties.

DISCUSSION

Mr. W. G. Bowman.[1]—I recently had occasion to see some bomber plant floors made of white cement and to discuss the question of their efficiency with some of the people in responsible charge.

Careful lighting tests made at the time the floors were built showed 36.5 foot candles on a horizontal plane and about 20 on a vertical plane, the latter being the more significant since it takes into account some of the reflectivity of the floor itself. Now after nine months use, the reflectivity on the vertical plane is reduced about 25 per cent.

In this same plant there are also gray cement floors. At the time they were built they showed about 16 foot candles on the vertical plane as compared with 20 for the white cement floors. In nine months the gray cement floors were down to 8 foot candles. Thus the gray cement floors lost 50 per cent and the white cement floors 25 per cent in nine months.

Although the white cement floor cost about 5 cents a square foot more than the gray, the owners seem satisfied, claiming that they have saved the difference in their light bills.

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[1] Editor, *Engineering News-Record*, New York, N. Y.

THE INFLUENCE OF THE DURABILITY OF AGGREGATE UPON THE DURABILITY OF THE RESULTING CONCRETE

By H. H. Munger[1]

Synopsis

The chief purpose of the investigation herein reported was to determine the extent to which the results of the soundness test of aggregate by the freezing-and-thawing method may be relied upon as a criterion of the durability of the resulting concrete.

Fourteen aggregates of very radically different physical and chemical properties were used in the construction of 2 by 2 by 11.4-in. concrete beams. Representative samples of each were tested for soundness by freezing and thawing. The beams were subjected to freezing and thawing, the dynamic modulus of elasticity being used as an index of progressive deterioration. The results are compared with field service records where available.

From time to time specimens were removed from the freezing-and-thawing series and tested in flexure and as modified cubes. The results of these tests are compared with the dynamic test results.

Data are presented that indicate:

1. There is no dependable correlation between results of the soundness test upon aggregate, *per se*, and the durability of the resulting concrete.
2. Thermal shock may be a much more important factor in laboratory tests than is generally recognized.
3. Durability tests should be made on concrete mixed in the proportions to be used in the field.
4. Field service records should be given greater weight than laboratory tests in acceptance or rejection of aggregates.
5. In all but one of the concretes investigated, the dynamic modulus of elasticity correlated very closely with the modulus of rupture, although the ratio was not a straight-line function.
6. Compressive strength is not a reliable index of deterioration in concretes of the type used in this investigation.

The search for a laboratory test that will make possible the accurate prediction of the durability of concrete made from any chosen aggregate has produced a considerable variety of procedures, some of which are more to be recommended for their adaptability to laboratory equipment and convenience than for their value as criteria of permanence of structures. Too much attention has been devoted to the search for *speed* in obtaining test results and too little to the *reliability* of the results.

Recently published reports of investigations in this field show that there is a general dissatisfaction with present test procedures and that there is ample cause for this dissatisfaction. Durability is perhaps the most important property of concrete. One of the greatest needs in the field of concrete engineering, therefore, is for some dependable means of predicting the durability of concrete of given proportions and materials when exposed to natural weathering.

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[1] Research Assistant, Department of Applied Mechanics, Kansas State College of Agriculture and Applied Science, Manhattan, Kans.

The influence of the water-cement ratio on the durability of the cement-water paste has been quite satisfactorily worked out, but the influence of the aggregate is not so clearly established because of the many overlapping and sometimes conflicting factors involved. Of all the soundness tests for aggregates now in use, that by actual freezing and thawing seems most closely to simulate natural weathering conditions, but even this test departs very widely from the processes of nature.

TABLE I.—GRADATION OF AGGREGATES.
Gradation Factor = 4.76.

Sieve Size	Proportional Parts	Percentage Retained	Accumulative Percentage
¾ in.	0	0.0	0.0
⅜ in.	1000	27.0	27.0
No. 4	750	20.3	47.3
No. 8	562	15.2	62.5
No. 14	422	11.4	73.9
No. 30	316	8.6	82.5
No. 50	237	6.4	88.9
No. 100	178	4.8	93.7
No. 200	134	3.6	97.3
Pan	100	2.7	100.0
Total	3699	100.0	

OUTLINE OF TESTS

In planning this investigation, every practicable means was adopted to eliminate all variables except those resulting from the varying characteristics of the aggregates. Because of the intricate interrelations of the different characteristics of concrete, it was found to be impossible to standardize all factors simultaneously.

All mineral aggregates were screened and recombined in a sieve ratio of 0.75; that is, for all sizes below the chosen maximum, the quantity of material retained on each sieve of the Standard Gradation Series (including the No. 200 sieve and the pan) was 75 per cent of that retained on the next coarser sieve. With 100 per cent passing the 0.5-in. sieve, this gives a gradation factor of 4.76. (See Table I.)

A water-cement ratio of 0.7 by volume was adopted to insure a paste of maximum durability, it being desired to produce a concrete in which the aggregate would be the least durable constituent.

Because of the widely differing characteristics of the aggregates selected, it was obvious that the use of a uniform mix, in all cases, would result in concretes of widely differing consistencies unless the quantity of mixing water was adjusted to compensate for the varying harshness of the aggregates.

It was necessary to choose between a constant mix and a constant workability. The latter was chosen because durability of concrete seems to be more directly influenced by workability than by mix. The quantity of water was varied only enough to compensate for the absorption of the aggregates. Even this was by no means simple or satisfactory. Natural rock is anything but homogeneous. No two samples will check exactly in absorption; the same sample, when redried, broken to smaller size, and retested will usually show higher apparent absorption and density. There is also doubt as to whether the absorbed water may not leave the aggregate during the setting of the concrete and become active in the hydration of the cement. Another element of uncertainty is the part played by the surface moisture on the aggregate. It remains to be proven whether this surface moisture, usually about 1 per cent of the weight of the aggregate, should be considered as active mixing water.

AGGREGATES

Series A—Ledge Flint.—A dark gray, rather porous, highly fossiliferous flint from the Schroyer limestone in the lower Permian. This rock characteristically breaks up into small rectangular fragments on exposure to weathering and has never been used commercially.

Series C—Cottonwood Falls Limestone, upper ledge.—A soft, light gray, highly absorptive, fossiliferous limestone near the base of the Permian system. This ledge forms a prominent outcrop in the lower levels of the Flint Hills, extending entirely across the state of Kansas and is characterized by its high resistance to weathering. This rock was formerly widely used as coarse aggregate and as building stone and gives excellent service in concrete but shows sloughing and spalling when dressed surfaces are exposed to weathering without good drainage.

Series D—Bala Serpentine.—A serpentinized peridotite consisting of serpentine, calcite, ilmenite, magnetite, phlogopite, spinel, and possibly other minor accessory minerals. This is a small dyke, of post-Permian age, outcropping near Bala, Kans., never used except experimentally.

Series H—Fort Riley Limestone.—A massive, light buff, Permian limestone, similar to the Cottonwood Falls limestone but somewhat softer; much used as a building stone, with satisfactory result.

Series J—Florence Limestone.—A soft, fossiliferous limestone containing 20 to 50 per cent of flint nodules; highly resistant to erosion; occurring immediately below the Fort Riley limestone; formerly used as aggregate in concrete pavements with good results.

Series K—Three-Mile Limestone (Formerly known as the lower beds of the Wreford Limestone).—A dense, cherty limestone of fairly high strength but relatively low resistance to weathering. It occurs about 150 ft. above the Cottonwood Falls limestone. It was formerly quarried commercially, but is no longer used because of unsatisfactory qualities.

Series M—Castle Rock Chalk.—An almost pure chalk from the upper portion of the Niobrora formation of the Cretacious System. It is characterized by exceptionally high absorption and low resistance to weathering in humid regions. Composed almost entirely of the microscopic remains of lime-shelled Foraminifera, it is occasionally used as building stone with good results, where not exposed to moisture.

Series P—Neva Limestone, unsound ledge.—A bluish-gray, soft, shaly limestone obtained from the waste heaps of quarries operating in the Neva; a limestone found about 35 ft. below the Cottonwood Falls Limestone. This ledge is regarded as entirely worthless and is wasted at the quarry.

Series S—Ness County Quartzite.—A very coarse-grained, hard, dense, heterogeneous rock of Tertiary age, consisting of quartz, feldspars, aragonite, and amphiboles, cemented with secondary silica (opal). In some places it approaches conglomerate in texture. It possesses extremely high resistance to weathering, and has been used on a few local construction jobs of recent date.

Series T—Lincoln Sandstone.—A very hard, dense, fine-grained sandstone (often called quartzite, but lacking some of the lithological properties of that rock) consisting chiefly of highly angular grains of quartz, cemented with calcite, and containing numerous small concretions of pyrite or marcasite. This rock, which is in the basal section (Dakota sandstone) of the Cretaceous system, has been quarried commercially for many years and possesses an exceptionally good service record in concrete of all classes except where exposed to acids.

TABLE II.—PHYSICAL PROPERTIES OF AGGREGATES USED.

Series	Bulk Specific Gravity	Absorption, per cent	Abrasion Test, per cent loss		Modulus of Elasticity, E, millions psi.			Compressive Strength, psi.	Soundness Test by Freezing and Thawing	
			Deval	Los Angeles	Compression	Flexure			Aggregate, Loss of Weight at 25 Cycles, per cent	Concrete, Loss of E at 100 Cycles, per cent
						Static	Dynamic			
A	2.34	4.6	4.2	16.8	[a]	[a]	[a]	[a]	8.8	41.4
C	2.21	6.0	7.5	40.5	5.5	5.7	4.5	8 400	1.3	5.9
D	2.40	4.0	4.1	21.9	6.1	4.3	6.1	7 600	18.2	7.7
H	2.24	5.5	8.6	35.9	4.6	[a]	4.4	3 900	0.1	10.5
J	2.60	5.8	11.2	42.1	3.1	[a]	3.6[b]	5 600	6.8	29.7
K	2.39	3.5	9.7	34.1	[a]	[a]	[a]	[a]	1.9	32.7
M	1.45	29.4	53.1	96.0	0.35	[a]	0.22	740	100.0	100.0
P	2.41	4.3	5.8	52.3	5.5	[a]	1.3	8 700	32.7	9.5
S	2.36	1.1	6.2	42.9	6.0	[a]	6.5	18 000	0	41.6
T	2.65	0.3	3.9	43.9	11.5	[a]	10.8	6 560	0.3	15.6
U	0.24	107.9	[a]	[a]	0.00016	[a]	[a]	Indefinite	0	72.3
MR	0.88	[a]	[a]	[a]	0.002[c]	[a]	[a]	[a]	0	5.0
GLS	2.49	0	[a]	[a]	[a]	10.5	9.8	[a]	0	29.3
SG	2.62	0.5	[a]	[a]	[c]	[c]	[c]	[c]	2.0	50.6

[a] Determination impracticable.

[b] Determined from limestone specimen. Aggregate contained approximately 50 per cent flint.

[c] Tensile modulus, pure latex, according to L. S. Marks, "Mechanical Engineering Handbook," p. 759, McGraw-Hill Book Co., Inc., New York, N. Y. (1930).

Series U—Ground Cork, of the type commonly used for thermal insulation, was adopted as an aggregate to determine the influence of exceptionally low elasticity and thermal conductivity. With this material it was necessary to use 34 per cent, by volume, of fine sand to supply the lack of the smaller sizes of cork.

Series MR—Mealorub.—An experimental grade of granulated pure latex, was used for the same reasons as those stated for the use of cork.

Series GLS—Common Glass.—Obtained by crushing bottles and other containers was adopted as an aggregate to demonstrate the effects of high density, elasticity, and thermal conductivity. This material was later found to be highly reactive with cement.

Series SG—Kaw River Sand-Gravel.—This material, obtained by pumping from the bed of the Kaw River, consists chiefly of quartz and feldspars with flint, chert, limestone and several accessory minerals present in minor quantities. It is extensively used in concrete, both as fine and as total aggregate, service records showing generally unsatisfactory results in the latter case.

The physical properties of these materials are shown in Table II. It will be noted that they include the widest obtainable variations in hardness, absorption, density, resistance to abrasion, and soundness.

Soundness of the aggregates was determined by the A.S.T.M. Tentative Method of Test for Soundness of Aggregates by Freezing and Thawing (C 137 – 38 T).[2]

CEMENT

The cement used was a brand of portland cement in common use throughout Kansas. It passed all requirements of the A.S.T.M. Standard Specifications for Portland Cement (C 150 – 41)[3] for type I cement. A quantity sufficient for all specimens to be made for this investigation was thoroughly mixed in a concrete mixer and stored in covered metal containers until used.

The chemical analysis was as follows:

Magnesium oxide (MgO), per cent	3.40	
Sodium oxide (Na_2O), per cent	0.26	Na_2O eq. = 0.64
Potassium oxide (K_2O), per cent	0.57	
Sulfur trioxide (SO_3), per cent	1.46	
Ignition loss, per cent	2.17	
Insoluble residue, per cent	0.16	

WATER

The mixing water used was distilled at atmospheric pressure and stored in glass containers until used.

PREPARATION OF SPECIMENS

A quantity of graded aggregate was prepared for each batch, slightly in excess of expected requirements. The absorption was determined from a representative sample, in accordance with the A.S.T.M. Standard Method of Test for Specific Gravity and Absorption of Course Aggregate (C 127 – 39).[4] A quantity of water was added to the aggregate, equal to the determined absorption and an additional quantity, equal to 1 per cent by weight of the aggregate. This was mixed in a metal pan, covered to reduce evaporation, and allowed to stand for 1 hr.

The required quantity of cement was placed in another metal pan, water was added in the ratio of 0.7 by volume, and mixed to an even-textured slurry. The saturated aggregate was then added by increments, each increment being mixed to a uniform consistency. As the mix neared the desired degree of workability the increments were made smaller and the consistency was tested by means of the flow table, using the A.S.T.M. Method of Test for Flow of Portland-Cement Concrete by Use of the Flow Table (C 124 – 39).[5] The final flow table value was held within the limits of 95 to 105 per cent except in the case of the glass aggregate which, because of its extreme angularity, would have required an excessively rich mix to produce such workability.

To eliminate variables resulting from insufficient manipulation, the minimum mixing time was set at 5 min. after the addition of the last increment. This resulted in a very uniform concrete, as was later proved by the generally close agreement of results from different specimens molded from the same batch.

Except in the case of Mealorub, only a very small quantity of which was available, a series of ten beams was molded from each of the 14 aggregates in a single batch. The molds were of welded steel plates, polished on the inside surfaces, and resting upon machined, cast iron pallets. The dimensions of the finished specimens were 2 by 2 by 11.4 in.

Molds and pallets were coated with a thin film of mineral grease before filling.

Immediately after molding, the specimens were placed in the moist room and protected from dripping water for 18 hr. The molds were then removed and moist storage continued to the age of 28 days. While not perfectly controlled, moist room temperatures were seldom outside of the range 70 to 75 F. for any appreci-

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[2] 1939 Book of A.S.T.M. Standards, Part II, p. 990.

[3] 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 1.

[4] 1939 Book of A.S.T.M. Standards, Part II, p. 300.

[5] *Ibid.*, p. 337.

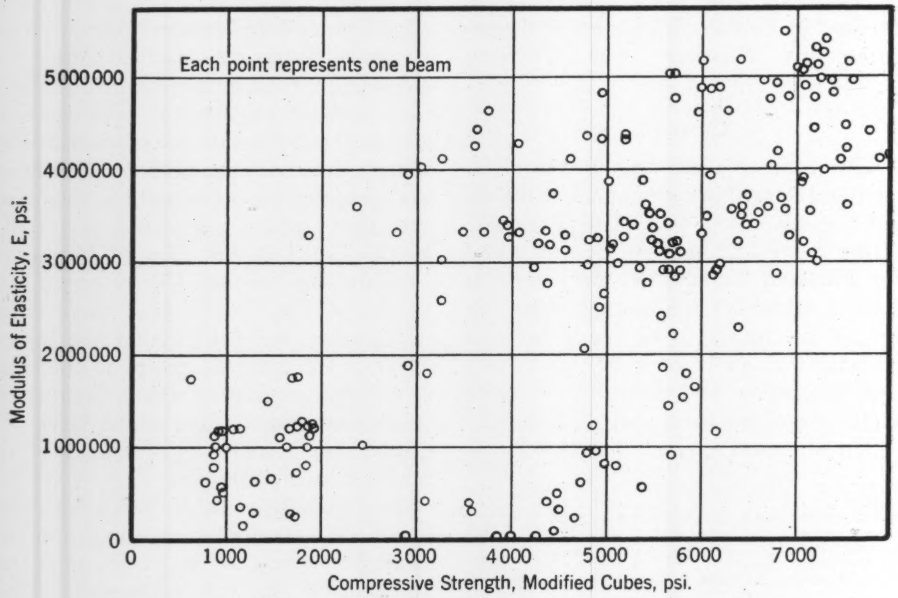

FIG. 1.—Relation of Modulus of Elasticity to Compressive Strength.

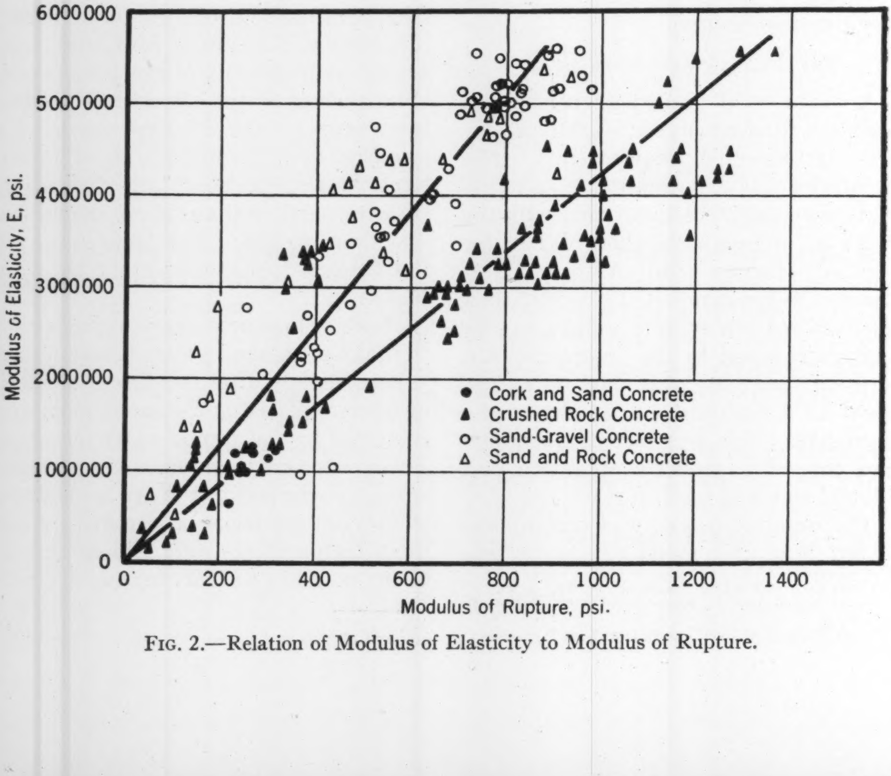

FIG. 2.—Relation of Modulus of Elasticity to Modulus of Rupture.

able length of time, and relative humidity was almost continuously above 95 per cent.

FREEZING-AND-THAWING TEST

At the age of 28 days the specimens were divided into two lots, one lot being held in moist room storage, the other subjected to freezing and thawing by the rapid change method, substantially as described in A.S.T.M. Method C 137, freezing to approximately −10 F. and thawing by placing in water at 80 F. The specimens were returned to the refrigerator after about 30 min. in the water. Usually four cycles per day were obtained, the specimens remaining in the frozen state over nights and holidays.

The dynamic modulus of elasticity was determined at intervals of about one week on both the moist room and the soundness test specimens.[6] From time to time (usually at intervals of 100 cycles) beams were taken from both lots and tested in flexure and as modified cubes.

TEST DATA

The test results are presented in the form of graphs, Figs. 1 to 16.

Figures 1 and 2 include data from several series of similar beams that received identical treatment with those reported in full, but did not give results of sufficient importance to merit space in the text of this paper.

Figure 1 shows the relation of compressive strength to modulus of elasticity. The correlation is obviously poor. Examination of broken beams showed that in most cases the cement paste was in almost perfect condition, the deterioration having been chiefly in bond or in the aggregate. When concrete of this nature is subjected to compression, the damaged aggregate is keyed together in the matrix of cement paste and the compressive strength is comparatively high.

In several cases, beams had so far deteriorated that they could be easily broken to bits with the bare hands, yet they showed compressive strength above 3000 psi.

The modulus of rupture, as shown in Fig. 2, shows much better correlation with modulus of elasticity. The reduction in tensile strength resulting from damage to bond or from deterioration of aggregate particles is reflected in proportionate reduction in modulus of rupture and modulus of elasticity. For each different variety of aggregate this is approximately a straight-line relation but different aggregates show different ratios of change in modulii.

For an investigation such as the one herein reported, the dynamic modulus of elasticity is almost ideal as an index of deterioration, since the same specimen may be tested any number of times under a great variety of conditions and its progressive changes definitely recorded without damage to the specimen.

Figures 3 to 16 show the change in dynamic modulus of elasticity for each of the 14 aggregates tested.

Ledge flint, series *A*, Fig. 3, concrete failed in bond and by splitting of aggregate particles, the fragments being hard, sharp, and apparently sound, the failure being of the same nature as when the stone is tested *per se*.

Cottonwood Falls limestone, series *C*, Fig. 4. Samples of freshly quarried rock from this ledge usually fail in soundness. Weathered samples, such as were used in this series are quite uniformly sound. This series showed the least damage of any mineral aggregate included in this investigation. Pavements and structures in which this rock was used as coarse aggregate are in

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[6] W. T. Thomson, "Measuring Changes in Physical Properties of Concrete by the Dynamic Method," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 1113 (1940).

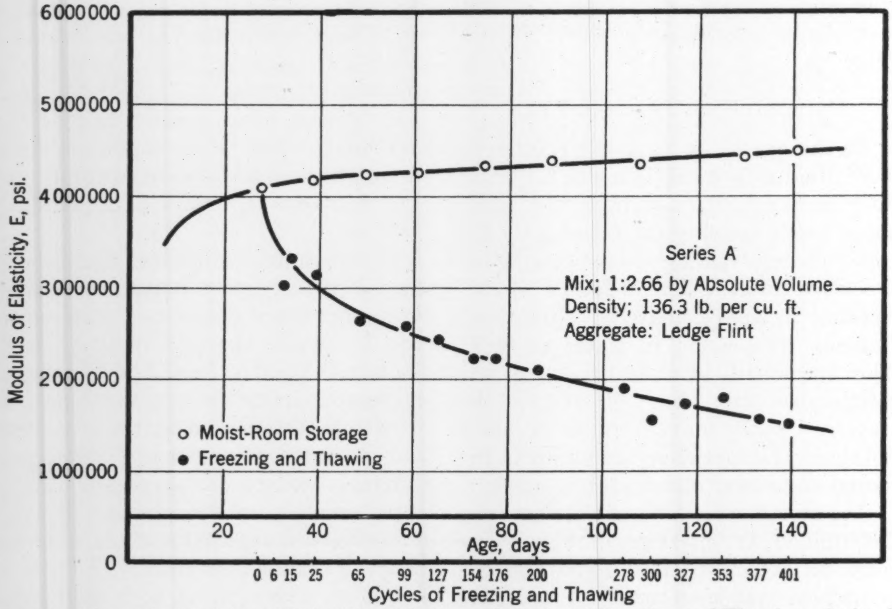

FIG. 3.—Change in Modulus of Elasticity with Age and Cycles of Freezing and Thawing—Series A.

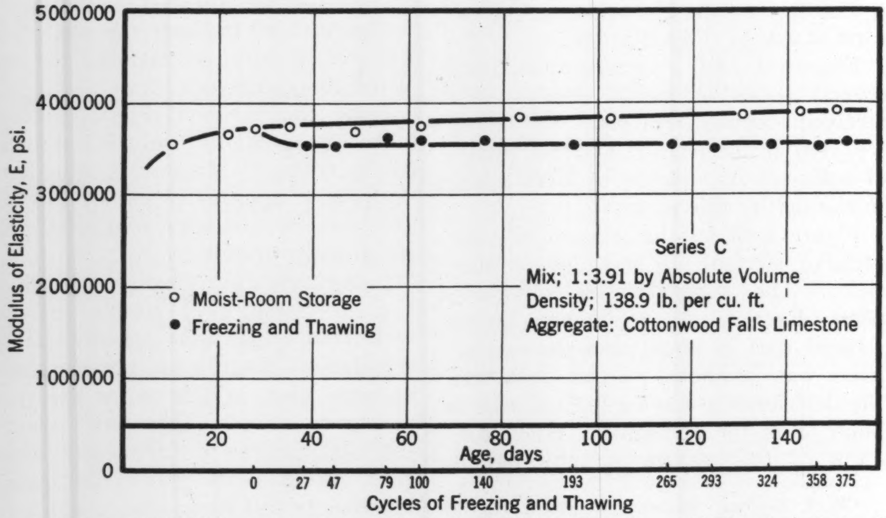

FIG. 4.—Change in Modulus of Elasticity with Age and Cycles of Freezing and Thawing—Series C.

excellent condition after 25 to 40 yr. service.

Bala serpentine, series *D*, Fig. 5, fails quickly in the soundness test as ordinarily performed, the rock splitting up into granules that seem to be governed in size and shape by the forms of the original crystals. However, when allowed to thaw in air at room temperature, the remainder of the cycle remaining unchanged, freezing and thawing have almost negligible effect. Apparently the damage results from the thermal stresses induced by rapid rise in temperature. In concrete, this proved to be the second best rock tested when rated according to percentage loss of elasticity. Experimental concrete from this rock, when exposed to the severest possible natural exposure, has shown no damage whatever in 2 yr.

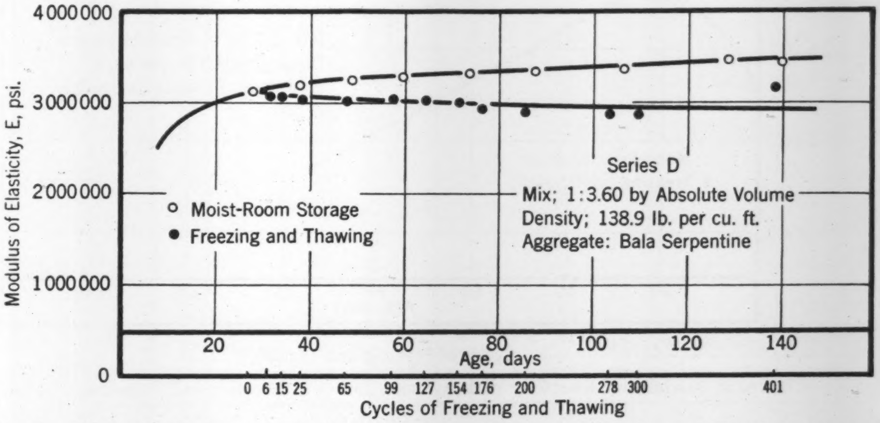

FIG. 5.—Change in Modulus of Elasticity with Age and Cycles of Freezing and Thawing—Series D.

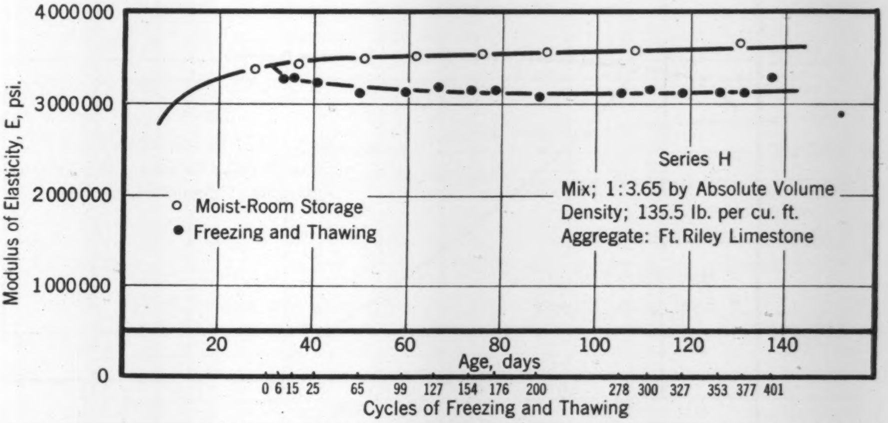

FIG. 6.—Change in Modulus of Elasticity with Age and Cycles of Freezing and Thawing—Series H.

Fort Riley limestone, series *H*, Fig. 6, has given uniformly satisfactory service in concrete and as building stone and withstands the soundness test with slight damage either in concrete or *per se*.

Florence limestone, series *J*, Fig. 7, has been condemned as unfit for use by the Materials Dept. of the Kansas State Highway Commission on the basis of laboratory tests. It fails by splitting of flint nodules but (like the Bala serpentine) shows relatively slight damage if allowed to thaw in air. Damage seems to be chiefly from thermal shock. Concrete pavement containing this rock as coarse aggregate has given 27 yr. of satisfactory service under highly unfavorable circumstances.

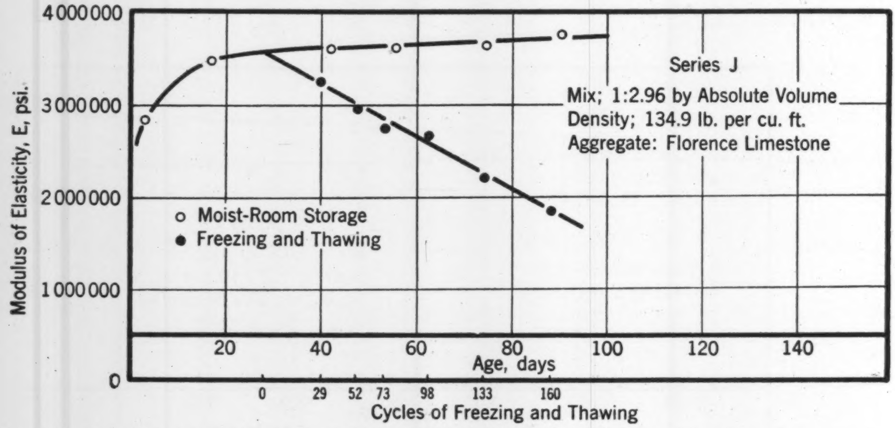

FIG. 7.—Change in Modulus of Elasticity with Age and Cycles of Freezing and Thawing—Series J.

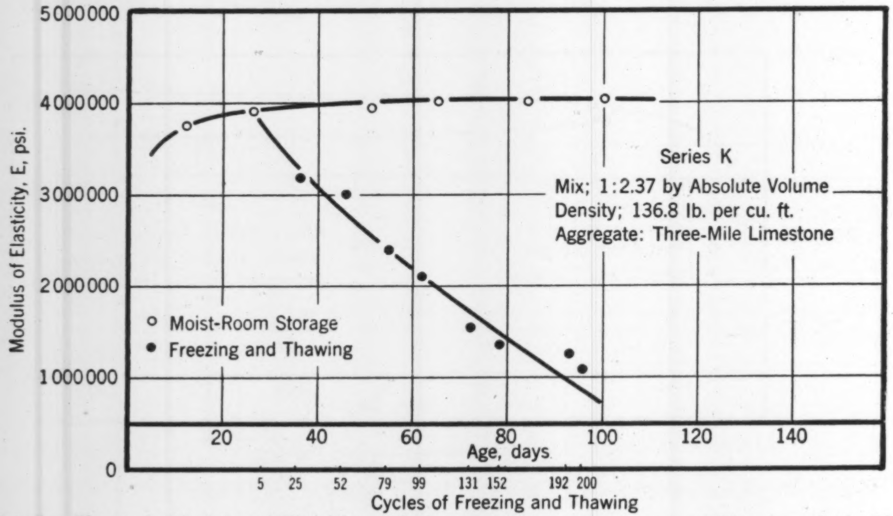

FIG. 8.—Change in Modulus of Elasticity with Age and Cycles of Freezing and Thawing—Series K.

Three-mile limestone, series *K*, Fig. 8, has never been extensively used in construction, and field service records are scattering and indefinite but seem to indicate unsatisfactory performance.

Samples from this ledge have been reported as acceptable by the Materials Testing Laboratory of the Kansas State Highway Commission. The loss in weight at 25 cycles was 1.9 per cent, placing this rock definitely in the satisfactory class, yet its concrete showed 32.7 per cent loss at 100 cycles and failed completely at 200 cycles.

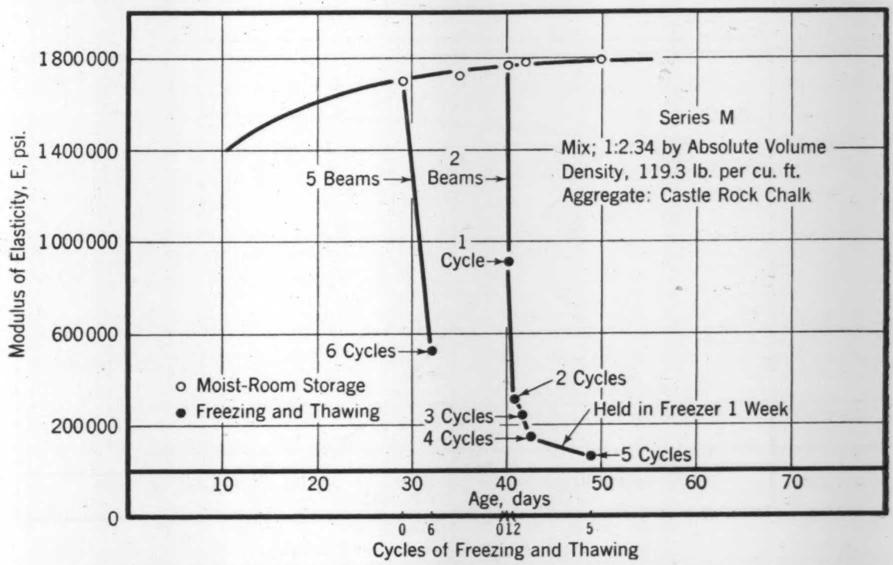

FIG. 9.—Change in Modulus of Elasticity with Age and Cycles of Freezing and Thawing—Series M.

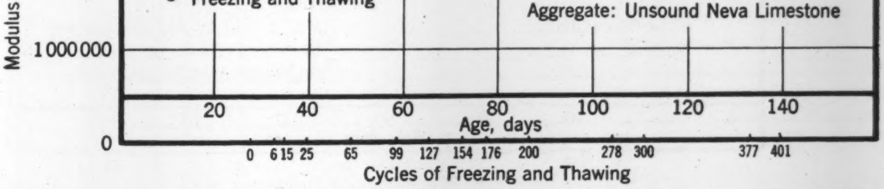

FIG. 10.—Change in Modulus of Elasticity with Age and Cycles of Freezing and Thawing—Series P.

Castle rock chalk, series *M*, Fig. 9, is known to be highly susceptible to attack by moisture. When not more than 50 per cent saturated it endures freezing and thawing with slight damage, but when saturated it disintegrates with considerable expansion within ten cycles. Test cylinders of identical composition with the beams of this series have withstood

2 yr. exposure to weathering with no visible damage when protected from ground moisture, but disintegrated within 3 months when placed in shallow water during winter weather.

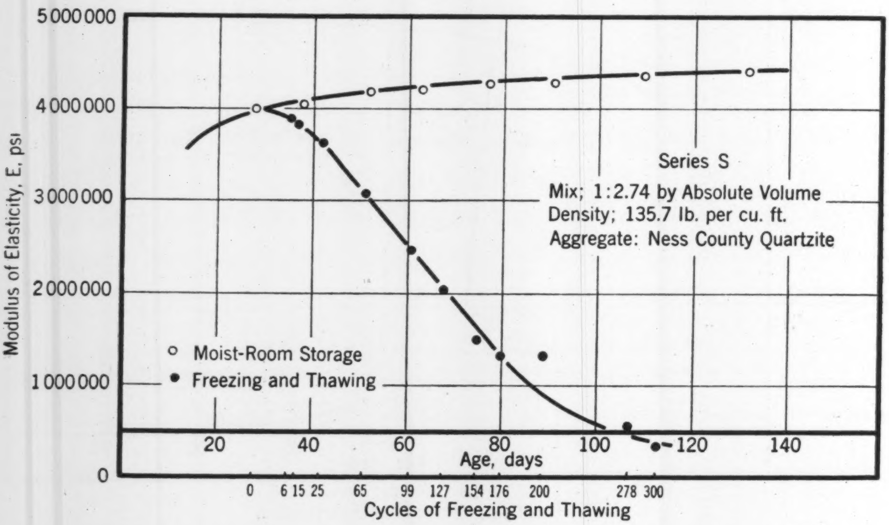

FIG. 11.—Change in Modulus of Elasticity with Age and Cycles of Freezing and Thawing—Series S

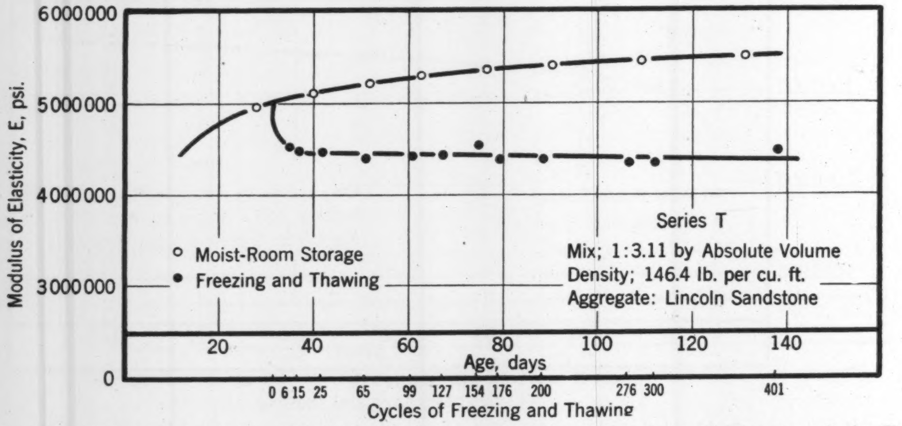

FIG. 12.—Change in Modulus of Elasticity with Age and Cycles of Freezing and Thawing—Series T.

Neva limestone, unsound ledge, series *P*, Fig. 10. This rock shows, in a striking manner, the importance of the cement paste as a protective covering. Where used in rich concretes it has given satisfactory service for as long as 35 yr. when the mortar coating remained unbroken, but it failed completely within 5 yr. when directly exposed to weathering. When the stone was tested *per se* it ranked ninth of the ten rocks investigated, yet its concrete ranks third. Where used in lean mixes this rock makes a concrete of very low durability.

Ness County quartzite, series *S*, Fig. 11,

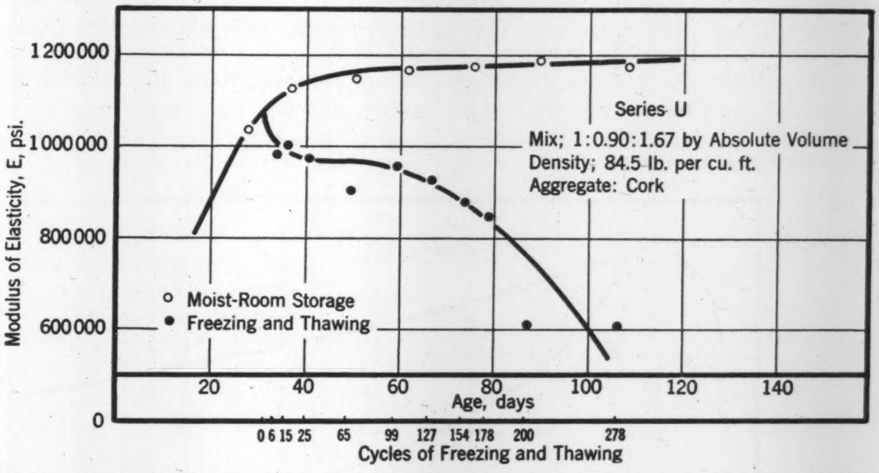

FIG. 13.—Change in Modulus of Elasticity with Age and Cycles of Freezing and Thawing—Series U

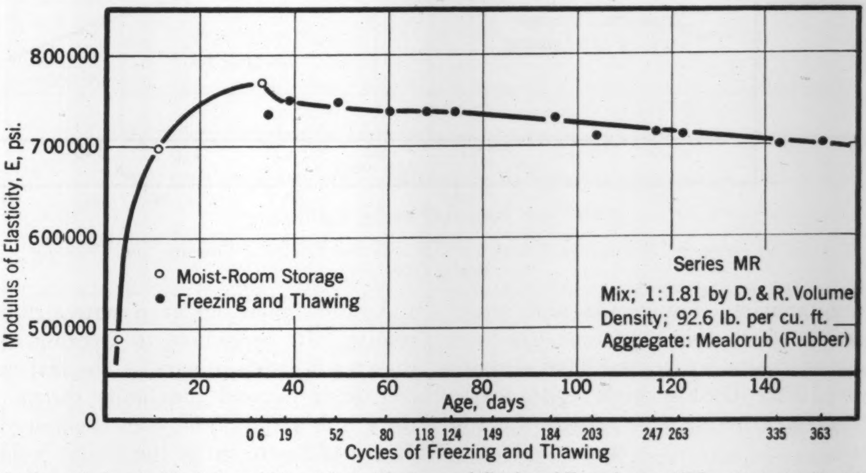

FIG. 14.—Change in Modulus of Elasticity with Age and Cycles of Freezing and Thawing—Series MR.

is notable for its extreme resistance to natural weathering as well as to laboratory tests. At 100 cycles it has shown no damage whatever, yet at 100 cycles its concrete had lost 41.6 per cent in elasticity, and at 300 cycles more than 90 per cent. Failure was in bond, neither the paste nor the aggregate showing any signs of damage and the compressive strength remaining above 3500 psi. It is possible that some chemical reaction may have contributed to this deterioration, although the fact that the moist room control specimens showed a consistent increase in elasticity up to the end of the period of observation seems to throw some doubt on that explanation. Stanton has shown[7] that opal may, under some conditions, become a reactive constituent in concrete and the cementing material of this rock is almost entirely opal and constitutes perhaps 25 per cent of the substance of the rock.

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[7] T. E. Stanton, "California Experience with the Expansion of Concrete Through Reaction Between Cement and Aggregate," *Journal*, Am. Concrete Inst., Vol. 38, No. 3, January, 1942, pp. 209–236.

Lincoln sandstone, series *T*, Fig. 12, has been tested up to 500 cycles, *per se*, with negligible loss in weight. Its concrete shows a loss of 10 to 15 per cent in elasticity within the first 15 cycles of freezing and thawing, after which practically no further loss occurs. No rational explanation has yet been found for these characteristics. Pavements and structures in which this sandstone has been used as coarse aggregate have demonstrated exceptionally high resistance to natural weathering over periods of many years.

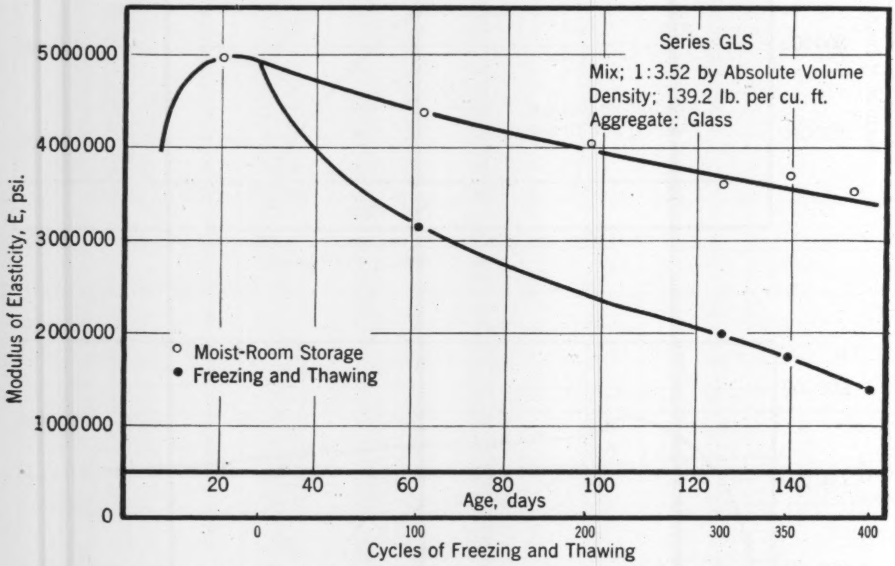

FIG. 15.—Change in Modulus of Elasticity with Age and Cycles of Freezing and Thawing—Series GLS.

Ground cork, series *U*, Fig. 13, as was expected, gave a concrete of low strength and very low elasticity but showed surprisingly high resistance to freezing. At 100 cycles no serious damage had been suffered. To appreciate fully the significance of this record, it may be compared with the performance of sample cores from State Highway pavements, many of which fail completely within the same number of identical cycles. At about 200 cycles the cork had decomposed to an earthy humus of considerably reduced volume. Water collected in the resulting voids, and failure appeared to be due to disruptive action of ice.

Mealorub, series *MR*, Fig. 14, possesses properties very similar to those of cork and gave similar but even more striking results. Its resistance to freezing and thawing is extraordinary. The test was continued beyond the limits shown in the graph and at 766 cycles the value of *E* was 42.2 per cent of the 28-day value, and, except for slight sloughing of the surface, the specimen had shown no visible signs of damage. Rapid deterioration became evident at about 800 cycles with almost complete failure at 990.

Common glass, series *GLS*, Fig. 15,

proved to be so highly reactive that in 150 days the moist-room specimens had lost 30 per cent of their 28-day elasticity, and the soundness test specimens in the same time, 400 cycles, had lost 70 per cent. The failure was in bond, accompanied by softening and expansion of the cement paste; the aggregate showed no indication of any damage whatever. It has been observed that where glass has been accidentally included in field concrete there is a tendency to develop "pop-outs" that include the glass fragments. This has sometimes been explained as being the result of the high elasticity, brittleness, poor bond, and high thermal expansion of glass. The results obtained in this investigation indicate chemical reaction as the probable cause.

Kaw River sand-gravel, series *SG*, Fig. 16, gave results in harmony with its usual performance in field construction. Deterioration was immediate and rapid and at 100 cycles amounted to approximately 50 per cent of the elasticity value of the moist room controls. Complete failure came before 400 cycles.

This sand-gravel is known to be reactive when used as total aggregate but gives completely satisfactory results when used with limestone coarse aggregate. The reactive mineral constituent has not yet been positively identified but data to date seem to indicate the soda-lime feldspars that are present in considerable percentages in the sizes between the 0.5-in. and No. 8 sieves.

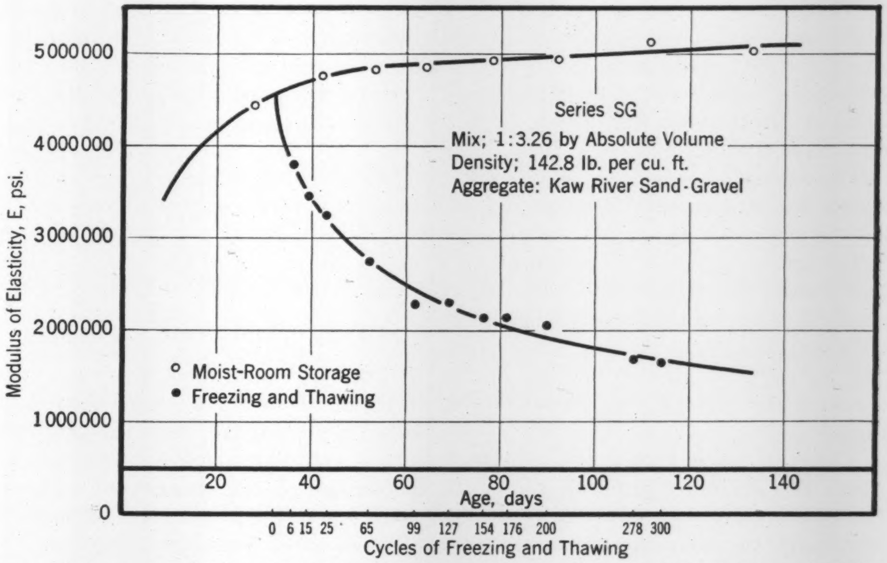

FIG. 16.—Change in Modulus of Elasticity with Age and Cycles of Freezing and Thawing—Series SG

INFLUENCE OF THERMAL SHOCK

Investigation of the influence of the thermal properties of aggregates has not progressed far enough to warrant any final conclusions, but a number of observed facts indicate the need for extensive research in that field.

Pickett[8] has demonstrated that it is possible to secure temperature stresses as great as 2600 psi. in a 3 by 6-in. cylindrical specimen during the thawing phase of the soundness cycle as ordinarily conducted. In the case of angular projections, such as are ordinarily found on aggregate samples, these stresses may be greatly increased.

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[8] G. Pickett, "The Effect of Biot's Modulus on Transient Thermal Stresses in Concrete Cylinders," *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 913 (1939).

Preliminary tests have shown that thawing in air at room temperatures reduces the damage to some rocks by more than 50 per cent. Concrete specimens show corresponding results. In this connection it is important to keep in mind that natural temperature changes rarely exceed 10 F. per hour, while the laboratory procedure gives changes as great as 100 F. in less than 1 min. on the surface of the specimen.

The work of Thomson[9] and Nothstine[10] shows that the thermal homogeneity of the concrete is a factor of considerable importance in the determination of soundness. In general it was found that the greater the difference in thermal diffusivity between the aggregate and the cement paste, the more liable is the concrete to damage by temperature change.

There is no evidence that thermal shock is a factor of any importance in the weathering of concrete in ordinary structures; therefore any laboratory procedure in which very rapid temperature changes are employed is subject to the suspicion that its results are not a dependable criterion of durability under natural weathering.

In this investigation, the aggregates that gave results showing the worst correlation with field results were those that are most affected by thermal shock. Further investigation along these lines is in progress with prospects of highly significant results.

FIELD SERVICE RECORDS

Because of the relative newness of laboratory tests for soundness, there is as yet a great lack of comparative dat on field exposure of concretes made fror tested materials.

In this investigation examination wa made of any pavements and structure known to have been constructed of th materials under study. In addition t this, the author has had more than 3 yr. experience with the use of some o these materials and observation of thei performance under field service conditions. A great mass of information ha thus been accumulated that cannot b included in a report of this nature because of space limitations. Conclusion 3 and 4 are based largely upon such fiel observations and are open to the objection that they are personal opinions They are defended upon the ground tha a quarter of a century of exposure t natural weathering is a more reliable test of the durability of any given material than is any accelerated laboratory test.

GENERAL DISCUSSION OF RESULTS

The behavior of these specimens under test calls attention to the well-known but often neglected fact that the properties of concrete depend less upon the intrinsic properties of the constituent materials than upon their mutual interrelations. This is most strikingly illustrated by series *D* and *S*. In the one case a rock that definitely fails to meet ordinary soundness specifications for aggregate forms a concrete that ranks far above the average in soundness. In the other case, a rock that resisted 200 cycles of freezing and thawing, with no indication whatever of any form of deterioration, formed a concrete that proved to be far below average in resistance to the same test. Too little is known of the nature of the changes taking place in concrete under weathering conditions to justify positive assertions as to the factors affecting durability, but it is highly significant that, in every case, rocks that have shown a high degree of resistance to natural weathering in concrete pavements and structures also show high resistance to laboratory freezing and thawing when tested in *concrete* specimens, but that no such agreement exists between field observations and laboratory tests of the aggregate, *per se*.

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[9] W. T. Thomson, "A Method of Measuring Thermal Diffusivity and Conductivity of Stone and Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 1073 (1940).

[10] L. V. Nothstine, "Thermal Diffusivity and Modulus of Elasticity in Relation to the Durability of Concrete." Unpublished thesis, Kansas State College (1940)

CONCLUSIONS

On the basis of data obtained in this investigation, and of results reported by previous investigators, the following conclusions are offered:

1. For materials of the types included in these tests, there is no dependable correlation between results of the soundness test upon aggregates, *per se*, and the durability of the resulting concrete.
2. Thermal shock may be a much more important factor in laboratory tests than is generally recognized, and is a subject requiring further investigation.
3. Durability tests should be made on concrete mixed in the proportions to be used in the field.[11]
4. Field service records should be given greater weight than laboratory tests in acceptance or rejection of aggregates.
5. In all but one of the concretes investigated, the dynamic modulus of elasticity correlates very closely with the modulus of rupture, although the ratio is not exactly a straight-line function.
6. Compressive strength is not a reliable index of deterioration in concretes of the type used in this investigation.

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[11] C. H. Scholer, "The Durability of Concrete," *Bulletin No. 31*, Engineering Experiment Station (1931).

DISCUSSION

Mr. A. T. Goldbeck.[1]—I seem to agree to some extent with what Mr. Munger has stated. We have been making various kinds of accelerated soundness tests on aggregates for a great many years and we have discovered in a number of cases that the accelerated soundness test does not always agree with service. Sometimes it will reject a perfectly good aggregate and sometimes it does not determine whether that aggregate is unsound when it actually is unsound in concrete.

I rather like the idea of determining the soundness of aggregate by determination of the soundness of the concrete itself. I think there are a number of matters which make for unsoundness in concrete which are connected with the aggregate but which at the same time are not determined by the soundness of the aggregate.

The question of bond has something to do with it. We have made some tests in connection with the shape of the roughness, if you want to call it that. Some aggregates have a particular type of fracture, a concoidal fracture, and when expansion and contraction take place in the mortar there is a tendency toward wedging action, a tendency to loosen the bond. Other aggregates have a rather precipitous fracture, a series of vertical faces so that when expansion and contraction of the mortar takes place, that expansion does not loosen the bond; the mortar simply expands or contracts against the vertical faces. All those things have to do with the question of soundness of concrete as influenced by the aggregate.

I think this matter of coefficient of thermal expansion is important too. Do not ask me whether an aggregate having a high coefficient of thermal expansion as compared with one of low thermal expansion is best. I do not know. There are some indications that a low thermal expansion has caused trouble at times, but on the other hand there are some indications that a low thermal coefficient of expansion may result in less cracking, of concrete roads, in particular. So there are a lot of things to find out. This is simply a start.

There is a joint research project in connection with the durability of concrete under way of which Mr. Scholer is chairman. We hope that project will look into the fundamental things in connection with durability. There are a lot of fundamental things—for instance, the question of what happens to water when it is in the pores of concrete or in the pores of the aggregate. Water does not freeze quite so readily when it is confined in these finely divided pores.

Mr. Fred C. Lang.[2]—Our experience a few years ago with deleterious materials in a gravel pit may be of interest. The specification under which the contract was awarded stated that there should not be over a certain percentage of shale or other material having similar characteristics.

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[1] Engineering Director, National Crushed Stone Assn., Inc., Washington, D. C.

[2] Engineer, Materials and Research, Highway Department Laboratory, Minnesota Highway Dept., University of Minnesota, Minneapolis, Minn.

In order to determine whether the other materials had similar characteristics, we made small concrete beams in which the coarse aggregate consisted entirely of one suspected type of deleterious substance. The concrete beams were subjected to freezing-and-thawing tests. After the testing was completed the contractor was invited to view the specimens. It was conclusively demonstrated to him that certain substances were similar in their characteristics to shale. The differences in the concrete-making properties of the aggregates did not all show up in the tests on the aggregates.

This and other work has made me question certain tests on aggregates. In general, I think the quality of aggregates should be determined by testing concrete in which they are used, keeping in mind the exposure to which the concrete is to be subjected.

MR. NICOLAAS T. F. STADTFELD.[3]—The City of New York Board of Water Supply is building 85 miles of concrete-lined tunnel for the Delaware Aqueduct, and naturally that means opening up a large number of virgin deposits in order to have the aggregates near the place where they are to be used. The difficulty is to write a specification which will fit aggregates for that long a stretch. Our specifications do not include the sodium sulfate test, the magnesium sulfate test, or freezing-and-thawing test, and, from what I have heard this morning, perhaps the omission of the latter is just as well. However, we had to have certain protections against deleterious elements; against shale in fine aggregate we devised a boiling test whereby a 100-g. sample is run through a standard set of sand sieves, collected and boiled in 500 ml. of water for 2 hr.; it is then rerun through the same sieves; it was found that if any fresh shale is present in that sand the boiling test will break it down and increase the number of fines. That is one point of interest.

Another point of interest relates to coarse aggregate. On the north bank of Rondout Creek near Lackawack, N. Y., a deposit was found which yielded hard siliceous boulders and gravel. This was developed by one of our contractors. On the basis of his experience another contractor set up an elaborate fine and coarse aggregate production plant on the south bank but the crushed boulders and gravel encountered there were of a much more friable nature. As a first precaution all grits between the $\frac{3}{8}$ and $\frac{1}{4}$-in. sieves were removed. Although excellent strengths were obtained in concrete cylinders, there was still some doubt as to the durability of this rather soft sand-stone aggregate. In talking this over with Mr. Walker he suggested the making of thin sections and microscopic examination of the cementitious material between the grains. This was done, and as a siliceous and not an argillaceous matrix was found to be present, considerable worry was lifted from my mind. A service record of 3 yr. indicates that the aggregate is doing well.

There is one more point in our specifications which I should mention, and that is that they differ somewhat from other specifications in regard to the sand grading required. We allow no more than 3 per cent passing the No. 100 sieve, which is contrary to the tendency today. We realize that it is. But it must be considered that in these virgin deposits it is not known just how the very fine particles will react. There are some clays that are very detrimental and there are other clays that are helpful to concrete; and there often is not time to find out which type is present, so that in setting up specifications for an 85-mile stretch we decided that by holding the fines down to 3 per cent passing the No. 100 sieve we could minimize trouble from excessively fine material coating the sand grains. Since permanence in our work is of paramount importance, much of it being well-nigh inaccessible and difficult to repair afterwards, the extra cost of a higher cement content to take care of the lack of fines in the sand seems well justified.

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[3] Division Engineer, New York City, Board of Water Supply, New York, N. Y.

MR. H. S. MATTIMORE.[4]—The subject of concrete and aggregate durability is a very important subject, so important that a word of caution should be given when considering the author's conclusion that no laboratory test could be used to predict the action of the material. So far as this individual research is concerned this may be so, but certainly we should not leave the impression that a material must be tried in service before a decision can be made relative to its durability.

Many papers have been given on aggregate soundness before this and other national technical societies. Some of these have shown direct relation between laboratory and service tests.

This leads to another thought regarding the testing of materials in general, especially natural materials like sand, stone and gravel. Some knowledge of sources is essential to interpret tests on small samples submitted. I refer to the degree which the sample is representative of the supply, the possibilities of change of materials in source during operation, and methods of production, etc. In other words, no laboratory is justified in recommending the use of any natural material unless it has knowledge of the source of supply. In many organizations a policy has been established that no tests are conducted on natural materials until a full description of the source is furnished by employes of the organization who will supervise the use of the material.

Tests for soundness are valuable in determining the durability of aggregates, providing the cause for unsoundness is also determined. Illustrating this point, consider a soundness test on fine aggregate of crushed gravel or stone. The grains or particles are sound, but are weakened by particle shape, that is, when crushed into thin splinters.

Let me say again that papers of this kind are extremely valuable but caution should be used so that the conclusions drawn will definitely state that such conclusions are applicable only to the materials tested and the conditions encountered in service.

MR. H. H. MUNGER[5] (*author's closure, by letter*).—An investigator may perhaps be pardoned for a feeling of apprehension when the results of his investigations compel him to present a report that runs directly contrary to generally accepted standards and procedures. However, in this instance the discussions by Mr. Goldbeck and Mr. Lang indicate that the apprehensions were uncalled for and that the inconsistencies in test results that prompted this investigation are not confined to Kansas aggregates.

Mr. Stadtfeldt's report on the use of thin sections is very interesting. We are using the same method at Kansas State College in identification of mineral constituents of aggregates and believe that it holds promise of valuable results. There seem to be two major difficulties to overcome before this method can be of general utility. The number of competent microscopists is very small, and we do not yet have a clear understanding of the part played by the various minerals in the deterioration of concrete. Perhaps the thin section method itself may be the means of overcoming the second difficulty.

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[4] Engineer of Materials, Pennsylvania State Highway Dept., Harrisburg, Pa.

[5] Research Assistant, Department of Applied Mechanics, Kansas State College of Agriculture and Applied Science, Manhattan, Kans.

Mr. Mattimore's concern lest laboratory testing be discredited is one with which every materials engineer may sympathize. If our tests do not give us a dependable means of predicting the properties of our concrete we are surely in a sad state. It seems to the author that it is imperative that we diligently re-examine our testing procedures and attempt to determine what modifications are required to enable us to duplicate in a few days or weeks the results of many years of exposure to natural agencies of destruction. When that is accomplished we shall have removed one of the most serious defects in our present methods. Until that is accomplished we cannot honestly say that materials testing, as applied to concrete, is based upon scientific principles.

POWDERED ALUMINUM AS AN ADMIXTURE TO CONCRETE

By Roy W. Carlson[1]

Synopsis

This paper deals with the properties of normal-weight concretes puffed almost imperceptibly after mixing by the presence of small amounts of aluminum powder added to the cement or aggregate. Concretes containing small amounts of the aluminum powder exhibit increased resistance to freezing and thawing in calcium chloride, improved bond strength to steel, and greater flexural strength. The compressive strength is decreased for most plastic concretes, but may be increased for harsh mixes and for any concrete confined so as to restrain the attempted slight expansion.

The rates of expansion of fresh concretes containing aluminum powder are shown to be different for various types of aluminum and for cements of various alkali contents.

When powdered metallic aluminum is added to a concrete mix, hydrogen gas is liberated within the concrete mass. The gas is believed to be the result of a reaction between the aluminum and the mixing water but the alkaline ions naturally present in fresh concrete are necessary to promote the reaction. On this basis, each gram of aluminum can be shown to release 1.35 liters of hydrogen gas at 70 F. and atmospheric pressure. The gas evolution begins soon after mixing but may continue for from one to several hours, depending upon such factors as fineness and purity of aluminum, alkalinity of cement, etc.

The familiar use of relatively large amounts of aluminum powder for producing light-weight concrete is not discussed herein, but only the use of very small amounts for improving or modifying the properties of normal-weight concrete. The possibility of beneficial effects of small additions of aluminum was not discovered accidentally, but was predicted in 1932 by K. P. Billner, who subsequently found that about 0.007 per cent of aluminum (by weight of cement) improved both the compressive strength and the bond to reinforcing steel.

When a review was made early in 1939 of unpublished test results on powdered aluminum as an admixture to concrete, apparent contradictions were found. Some observers had obtained fairly consistent increases in compressive strength of about 25 per cent from the addition of 0.007 per cent of aluminum, while others had obtained decreases of about half this amount under supposedly similar conditions. The present paper appears to explain the discrepancies. It is based on the results of tests at the Massachusetts Institute of Technology on about 1000 specimens of various types and sizes. Also reported are the effects of several grades of aluminum powder on other properties as well as compressive strength. Special attention is given to the effects of aluminum powder on durability of concrete, bond to reinforcing steel, and flexural strength.

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[1] Associate Professor of Civil Engineering, Massachusetts Institute of Technology, Cambridge, Mass.

COMPRESSIVE STRENGTH

In an attempt to explain the contradictory results from using powdered aluminum as an admixture, several hundred concrete cylinders were tested for compressive strength. One-half of the number contained a small amount of aluminum powder, usually 0.007 per cent by weight of cement. The intention was to explore miscellaneous conditions to learn where the aluminum might be beneficial and where it might be detrimental. The several hundred specimens comprised a variety of cements, aggregates, gradations, curing conditions, pressures, times of placing, finenesses of aluminum, etc.

In general, the compressive strength of the concrete was affected only slightly by the small amount of aluminum powder. Later tests on bond to reinforcing steel, durability, and flexural strength indicated that concrete might be improved greatly without having its compressive strength raised. Therefore, no emphasis is placed on most of the compression tests. The average strength of all specimens containing aluminum was slightly higher than that of the controls, principally because a number of abnormal concretes containing aluminum exhibited unusually high strength. Under most of the conditions investigated, there was an appreciable reduction in strength, while under a few other conditions there was a rather large increase in strength. When the concrete mix contained crushed aggregate and was lean and very harsh, the aluminum powder caused increases in strength sometimes exceeding 25 per cent. The aluminum increased the strength also when the concrete containing aluminum was confined so as to prevent free expansion and thus produce internal pressure during setting. The tests were not planned to show whether the effect of aluminum powder was related to the setting time and bleeding characteristics of the cement.

The more beneficial effect of aluminum powder on harsher mixes is shown clearly in Fig. 1. These curves were obtained by testing a number of concretes with progressively coarser aggregate gradations, but with the consistency (slump) and the cement content held constant. The aggregate was crushed dolomite of $\frac{3}{4}$-in. maximum size throughout. Beginning with oversanded mixes and proceeding by steps toward harsher mixes, the water-cement ratio (unaffected by aluminum) was reduced by steps to maintain the chosen slump. In the range of plastic mixes, the compressive strength increased in accordance with the reduced water-cement ratio, but as the mix tended to become very coarse, full benefit of the reduced water-cement ratio was no longer obtained and strengths were lower. Thus, each curve shows a maxi-

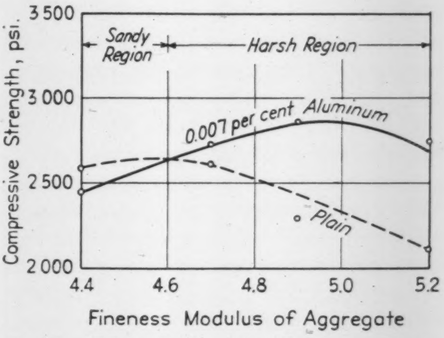


FIG. 1.—Effect of Aluminum Powder on Compressive Strength of Concrete for Various Gradations

mum strength for some particular fineness modulus, or in other words, for some particular degree of aggregate coarseness. In the concretes containing aluminum powder, the maximum strength is obtained with consistently coarser mixes (higher fineness moduli). It is apparent from the curves in Fig. 1 that if the effect of aluminum powder on compressive strength is investigated through using good quality, plastic mixes, one is likely to find aluminum to be detrimental. For harsh, unworkable mixes, on the other hand, the aluminum is likely to cause increased strength. The curves of Fig. 1 represent but one of several similar series of tests, all showing higher strength due to aluminum in the harsher mixes.

The benefit from aluminum powder only in coarse mixes may appear to have little practical value, except when the common occurrence of segregation is considered. In the segregated concrete without aluminum, the sandy portions are potentially strong and good, while the rocky portions are potentially weak and poor. In segregated concrete containing aluminum powder, it is expected that the sandy portions have their otherwise high strength reduced somewhat, while the rocky portions have their otherwise low strength raised markedly. The net result should be a considerable improvement in uniformity with aluminum. But regardless of what benefit aluminum powder can provide, every possible effort should be made to avoid segregation and to produce uniform concrete by natural means. The use of any material for no other reason than to permit poor workmanship is likely to be unsound.

The increased strength due to aluminum in harsh mixes of concrete may be considered merely as an elimination of the usual reduced strength of harsh mixes. In general, harsh mixes without aluminum develop considerably less strength than is predicted from the water-cement ratios. It is likely that the larger stones in harsh mixes are imperfectly cushioned in mortar, with the result that high concentrations of stress develop during loading, to cause premature failure. The effect of the aluminum may then be to expand the mortar slightly during setting and thus restore the cushioning of the larger stones. In tests for properties other than compressive strength (discussed below), the benefit from aluminum seems to be more directly related to elimination of bleeding and water gain, with the consequent better bonding of mortar to all sides of aggregate particles and to reinforcing steel. Although the aluminum powder does not improve compressive strength except in harsh mixes, it may improve other properties even in plastic mixes.

The optimum amount of aluminum powder for developing high compressive strength in concrete varies from about 0.003 per cent to 0.010 per cent by weight of cement. In plastic and rich mixes, reduction in strength is avoided only by using small amounts, while in harsh and lean mixes, increased strength can be obtained with higher percentages. In one series of tests on harsh concretes, where the mix was 1:6.7 by weight, highest strength was obtained with 0.010 per cent of aluminum, but 0.003 per cent produced almost as good strength and well above that of the control concrete. It is only for convenience that the quantity of aluminum is referred to the cement rather than to the volume of concrete, because it is usually the amount of gas evolution per unit volume of concrete that is important. Therefore, it should be recognized that a smaller percentage of aluminum should be used in rich mixes.

FLEXURAL STRENGTH

Flexural strength of concrete appears to be more responsive to benefit from aluminum powder than is compressive strength. Thus far, no large series of tests has been made to show the effect of optimum amounts of aluminum on flexural strength, but a number of exploratory comparisons were made and all showed increased strength due to the addition of about 0.007 per cent of aluminum. It was notable that even those concretes whose *compressive* strengths were affected adversely by the addition of aluminum powder, had their *flexural* strengths improved appreciably, often by 10 per cent or more.

TABLE I.—EFFECT OF ALUMINUM POWDER ON FLEXURAL STRENGTH OF CONCRETE BEAMS CAST ON END.

Distinguishing Feature	Average Modulus of Rupture, psi.		
Aluminum admixture, per cent	None	0.007	0.014
Coarse cement, 1380 sq. cm. per g	645	780	775
Fine cement, 1880 sq. cm. per g	762	1010	830

In order to explore the possibility that the improvement in flexural strength might be due to elimination of water gain and consequent better bond between paste and aggregate, a number of concrete beams were cast on end. In this way, those sides of the aggregate particles that were the undersides during setting were in the plane of a break when the beams were laid down for test. Two percentages of aluminum were tried with two finenesses of cement. Large increases in flexural strength were found due to the aluminum with both percentages of aluminum and with both cements, as may be seen in Table I. The larger amount of aluminum powder used (0.014 per cent) was enough to cause a decrease in compressive strength of about 20 per cent in similar concretes, and yet the flexural strengths were increased by as much as 9 and 20 per cent for the fine and coarse cement, respectively. The beams were small, only 2.5 by 2.5 by 10 in., but were made of plastic concrete containing natural aggregates of $\frac{3}{4}$-in. maximum size. Four beams were tested to obtain each average shown in Table I. The data are not complete enough to reveal the exact optimum percentage of aluminum powder for flexural strength but the high strengths obtained with 0.007 per cent indicate that the optimum is in this region for the 1:6 mix employed.

DURABILITY OF CONCRETE

In view of the recent discoveries that resistance to freezing and thawing of concrete may be improved by air entrained during mixing, tests were made to determine the corresponding effect of gas voids from aluminum powder. For this purpose, somewhat larger amounts of aluminum were added so as to decrease the weight of the concrete from one to several per cent. A very finely ground aluminum was compared with a coarser grade, and special cements were made to show the effect of intergrinding the aluminum with clinker. Also, some of the finer aluminum powder was preground with silica sand for promoting better dispersion in the concrete mix. Many other comparisons were planned that will become evident from the descriptions of the concrete mixes in Table II. It may be noted that the mixes comprise 3 different cements, 2 aggregates, 2 finenesses of aluminum, 4 different amounts of aluminum, and 3 ways of adding the aluminum.

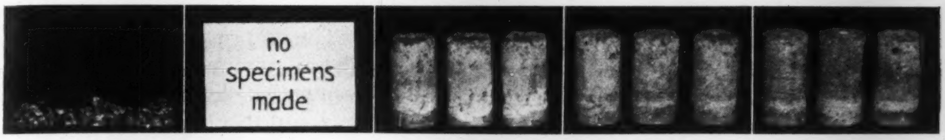
The durability tests included three types of test on concretes, (*a*) straight freezing and thawing, (*b*) freezing and thawing in calcium chloride solution, and (*c*) partial immersion in sodium sulfate solution. The results of the straight freezing-and-thawing tests can be reported in the single statement that only slight improvement was found due to the aluminum admixture. The sodium sulfate tests are not yet concluded, but they show no striking effect from the aluminum powder after many months. The more severe tests of freezing and thawing with calcium chloride show important differences and are therefore reported in more detail.

TABLE II.—DESCRIPTION OF CONCRETE MIXES FOR SPECIMENS TO SHOW EFFECTS OF ALUMINUM POWDER.

Mix	Aluminum Content, per cent	Fineness of Aluminum	Fineness Modulus of Combined Aggregate
	HARSH MIX, SILICEOUS SAND AND GRAVEL, MEDIUM ALKALI CEMENT		
No. 1......	None		5.64
No. 2......	0.007—interground with clinker	420 mesh (fine)	
No. 3......	0.014—interground with clinker	420 mesh (fine)	
No. 4......	0.028—interground with clinker	420 mesh (fine)	
No. 5......	0.014—interground with clinker	160 mesh (coarse)	
No. 6......	0.014—admixed to concrete	420 mesh (fine)	
No. 7......	0.014—admixed to concrete	160 mesh (coarse)	
No. 8......	0.014—preground with sand	420 mesh (fine)	
	PLASTIC MIX, SILICEOUS SAND AND GRAVEL, MEDIUM ALKALI CEMENT		
No. 9......	None		5.01
No. 10......	0.007—interground with clinker	420 mesh (fine)	
No. 11......	0.014—interground with clinker	420 mesh (fine)	
No. 12......	0.028—interground with clinker	420 mesh (fine)	
No. 13......	0.014—interground with clinker	160 mesh (coarse)	
No. 14......	0.014—admixed to concrete	420 mesh (fine)	
No. 15......	0.014—admixed to concrete	160 mesh (coarse)	
No. 16......	0.014—preground with sand	420 mesh (fine)	
	PLASTIC MIX, SILICEOUS SAND AND GRAVEL, LOW ALKALI CEMENT		
No. 17......	None		5.33
No. 18......	0.014—interground with clinker	420 mesh (fine)	
No. 19......	0.028—interground with clinker	420 mesh (fine)	
No. 20......	0.056—interground with clinker	420 mesh (fine)	
No. 21......	0.007—admixed to concrete	420 mesh (fine)	
No. 22......	0.014—admixed to concrete	420 mesh (fine)	
	PLASTIC MIX, SILICEOUS SAND AND GRAVEL, HIGH ALKALI CEMENT		
No. 23......	None		5.33
No. 24......	0.028—interground with clinker	420 mesh (fine)	
	PLASTIC MIX, CRUSHED DOLOMITIC AGGREGATE, LOW ALKALI CEMENT		
No. 25......	None		5.00
No. 26......	0.014—interground with clinker	420 mesh (fine)	
No. 27......	0.007—admixed to concrete	420 mesh (fine)	
No. 28......	0.014—admixed to concrete	420 mesh (fine)	

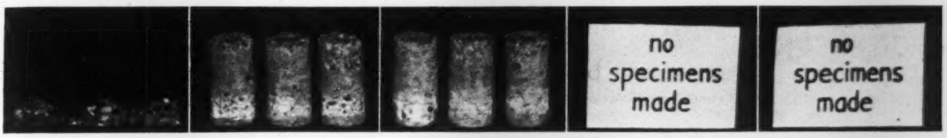
NOTE.—Mix 1:6 by weight throughout; $W/c = 0.54$ by weight for all mixes save Nos. 25 to 28 for which $W/c = 0.66$; slump approximately 4 in. throughout.

The procedure for freezing and thawing in calcium chloride was as follows: Cylinders of the concretes listed in Table II were first moist cured for 14 days. They were then alternately frozen and thawed once each day for 75 or more cycles. The thawing was by immersion to half depth in a 10 per cent solution of calcium chloride at 70 F. The specimens were precooled to about 30 F. while still in the solution, before being fully exposed to air at 0 F. overnight, the intention being to minimize


no specimens made

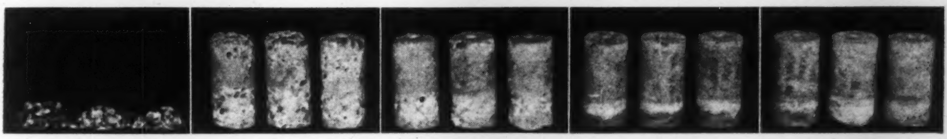
No aluminum Mix 17 | 0.007% aluminum | 0.014% aluminum Mix 18 | 0.028% aluminum Mix 19 | 0.056% aluminum Mix 20

Low Alkali Cement, effect of aluminum interground with cement.


no specimens made
no specimens made

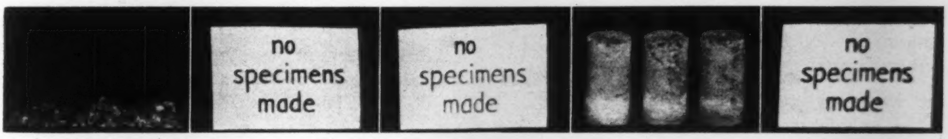
No aluminum Mix 17 | 0.007% aluminum Mix 21 | 0.014% aluminum Mix 22 | 0.028% aluminum | 0.056% aluminum

Low Alkali Cement, effect of aluminum added to mix.



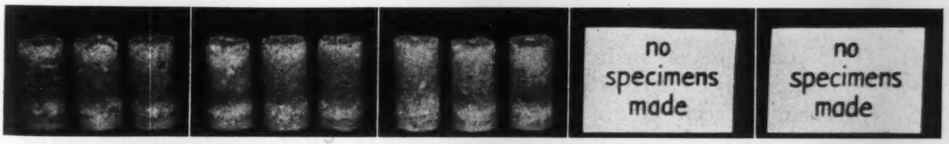
No aluminum Mix 9 | 0.007% aluminum Mix 10 | 0.014% aluminum Mix 11 | 0.028% aluminum Mix 12 | 0.056% aluminum No mix number

Medium Alkali Cement, effect of aluminum interground with cement.


no specimens made
no specimens made
no specimens made

No aluminum Mix 23 | 0.007% aluminum | 0.014% aluminum | 0.028% aluminum Mix 24 | 0.056% aluminum

High Alkali Cement, effect of aluminum interground with cement.


no specimens made
no specimens made

No aluminum Mix 25 | 0.007% aluminum Mix 27 | 0.014% aluminum Mix 28 | 0.028% aluminum | 0.056% aluminum

Low Alkali Cement, Crushed Aggregate, effect of aluminun added to mix.

FIG. 2.—Condition of Freezing-and-Thawing Specimens After 75 cycles.

drainage and drying from specimens before freezing. Length changes were observed periodically.

The actual length changes of specimens in the freezing-and-thawing tests are shown for 30 and 75 cycles in Table III. The condition of many of the more important specimens after about 75 cycles can be seen in Fig. 2. Invariably, the aluminum powder caused large improvements in resistance to freezing and thawing with calcium chloride, the improvements being greatest for the largest amounts of aluminum used (0.056 per cent interground with cement). The improvement was good whether the aluminum was interground with the cement or simply added to the concrete mix, but it was indicated that a given improvement could be obtained with less reduction in concrete weight when the aluminum was interground with the cement. The aluminum seemed to lose a considerable amount of its gas-generating power, but not so much of its effectiveness otherwise, after being interground with the cement. This is indicated by the results in Table III.

TABLE III.—EFFECT OF ALUMINUM POWDER ON DURABILITY AND OTHER PHYSICAL PROPERTIES OF CONCRETE.

Mix	Aluminum Content, per cent	Air Voids, per cent	Compressive Strength, psi.	Flexural Strength, psi.	Modulus of Elasticity, psi.	Freezing-and-Thawing Expansion, per cent	
						After 30 cycles	After 75 cycles
HARSH MIX, SILICEOUS SAND AND GRAVEL, MEDIUM ALKALI CEMENT							
No. 1	None	0.9	4240	690	5 350 000	0.110	(failed)
No. 2	0.007—interground	0.9	4800	680	5 350 000	0.011	0.250
No. 3	0.014—interground	1.4	4800	680	5 250 000	0.004	0.100
No. 4	0.028—interground	3.1	4780	780	5 100 000	0.002	0.070
No. 5	0.014—interground	2.6	2920	740	4 650 000	0.003	0.080
No. 6	0.014—admixed	4.3	3220	735	4 650 000	0.001	0.050
No. 7	0.014—admixed	3.1	2180	760	4 700 000	0.001	0.080
No. 8	0.014—admixed	4.2	3400	740	4 650 000	0.001	0.080
PLASTIC MIX, SILICEOUS SAND AND GRAVEL, MEDIUM ALKALI CEMENT							
No. 9	None	0.6	5090	800	5 300 000	0.060	(failed)
No. 10	0.007—interground	0.6	4640	780	5 300 000	0.007	0.035
No. 11	0.014—interground	1.2	4460	760	5 250 000	0.003	0.009
No. 12	0.028—interground	2.6	4660	800	5 100 000	0.000	0.004
No. 13	0.014—interground	2.3	2370	770	5 100 000	0.003	0.008
No. 14	0.014—admixed	4.6	3300	680	4 450 000	0.001	0.006
No. 15	0.014—admixed	4.6	2060	620	4 400 000	0.001	0.013
No. 16	0.014—admixed	4.3	2970	670	4 500 000	0.000	0.011
PLASTIC MIX, SILICEOUS SAND AND GRAVEL, LOW ALKALI CEMENT							
No. 17	None	0.7	5250	795	5 360 000	0.040	(failed)
No. 18	0.014—interground	3.2	4660	855	5 180 000	0.005	0.012
No. 19	0.028—interground	6.3	4210	795	4 350 000	0.006	0.014
No. 20	0.056—interground	10.8	2970	750	3 850 000	0.003	0.005
No. 21	0.007—admixed	1.9	4390	885	5 250 000	0.008	0.040
No. 22	0.014—admixed	3.7	3350	860	5 020 000	0.005	0.013
PLASTIC MIX, SILICEOUS SAND AND GRAVEL, HIGH ALKALI CEMENT							
No. 23	None	0.9	5400	805	5 360 000	0.070	(failed)
No. 24	0.028—interground	5.8	3500	760	4 780 000	0.003	0.010
PLASTIC MIX, CRUSHED DOLOMITIC AGGREGATE, LOW ALKALI CEMENT							
No. 25	None	1.0	4700	855	4 700 000	0.040	0.110
No. 26	0.014—interground	3.0	3720	...	....	0.016	0.035
No. 27	0.007—admixed	2.0	4100	900	4 700 000	0.013	0.031
No. 28	0.014—admixed	3.2	2800	900	4 500 000	0.009	0.024

In a typical case, 0.014 per cent of aluminum when admixed in the concrete produced 4.3 per cent of air (gas), while the same amount when interground produced only 1.4 per cent, but the latter gave excellent performance in the durability tests. Most of the loss of aerating power of the aluminum appeared to occur soon after grinding, with only a slow and gradual loss thereafter. The loss in aerating power of the interground aluminum powder was most severe for high-alkali cements. The concretes reported here were made at various ages after the cements were ground, but mostly after several weeks.

All of the control concretes reported in Table III failed completely in the freezing-and-thawing tests before 75 cycles, except that containing crushed aggregate. But even this concrete exhibited appreciable expansion, as compared with much less expansion for the corresponding concretes containing aluminum. This result should not be taken to prove that broken aggregate is necessarily better than natural aggregate, because the particular natural aggregate used contained a considerable amount of slate and was therefore not representative of the best natural aggregate.

BOND TO REINFORCING STEEL

A wide variety of tests on bond to reinforcing steel all show a large improvement in bond strength due to the addition of a small amount of aluminum powder. The specific concretes listed in Tables II and III were not tested for bond strength, because facilities were not available for making tests with aggregate as large as $\frac{3}{4}$ in. But many similar concretes employing the same cements were tested with $\frac{3}{8}$-in. maximum size of aggregate, and the results of the more important of these tests are shown in Table IV. Referring to the table, some improvement in bond strength is shown by every concrete containing aluminum, and in some cases the increase in bond strength is nearly 100 per cent. The particular type of bond test employed gave rather large variations and therefore no fine comparisons should be attempted from Table IV. Tests of a much more comprehensive nature are necessary to establish the relative merits of different grades of aluminum and of the method of incorporating it. The main value of Table IV is to show the general improvement in bond strength due to aluminum powder. Corresponding tests on the same cements interground with resin resulted in much lower bond strengths. These results are not included because more comprehensive results on the effect of resin are available elsewhere.

TABLE IV.—EFFECT OF ALUMINUM ADMIXTURE ON BOND TO REINFORCING STEEL.

Distinguishing Feature of Concrete	Bond Strength, Based on Total Area, psi.
Medium Alkali Cement:	
No aluminum	175
+0.014% fine aluminum, admixed	285
+0.014% coarse aluminum, admixed	255
+0.014% fine aluminum, preground with sand	325
+0.014% fine aluminum, interground with cement	365
+0.014% coarse aluminum, interground with cement	270
Low Alkali Cement:	
No aluminum	180
+0.007% fine aluminum, admixed	295
+0.014% fine aluminum, admixed	260
+0.014% fine aluminum, interground with cement	190
High Alkali Cement:	
No aluminum	140
+0.028% fine aluminum, interground with cement	245

NOTE.—Concrete was plastic mix, 1:5 by weight, natural aggregate of 3/8-in. maximum size, each specimen a 4-in. cube with 3/4-in. round rod passing horizontally through center, bond strength determined by "push-out."

MODULUS OF ELASTICITY

The increased porosity due to the action of aluminum powder in concrete is reflected in somewhat lower modulus of elasticity, E. The reduction in E was found to be negligible when the aluminum content was low enough to cause a reduction in concrete weight of less than 2 per cent, the general condition for optimum strength, but for larger amounts of aluminum, the reduction in E was from 2 to 4 times as great, percentagewise, as the reduction in concrete weight. Measured values of E are included in Table III for all of the concretes listed in Table II.

RATE OF GAS LIBERATION FROM ALUMINUM IN CONCRETE

The rate at which gas is liberated by the action of aluminum powder in concrete is of some importance. Aside from the fact that it would seem to be desirable for the action to go on after the concrete is placed, the effectiveness of the aluminum probably depends somewhat on how the time of gas evolution compares with the setting time of the cement.

A number of tests were made to show the rate of gas liberation, as measured by expansion of the fresh concrete, for a variety of conditions. The apparatus

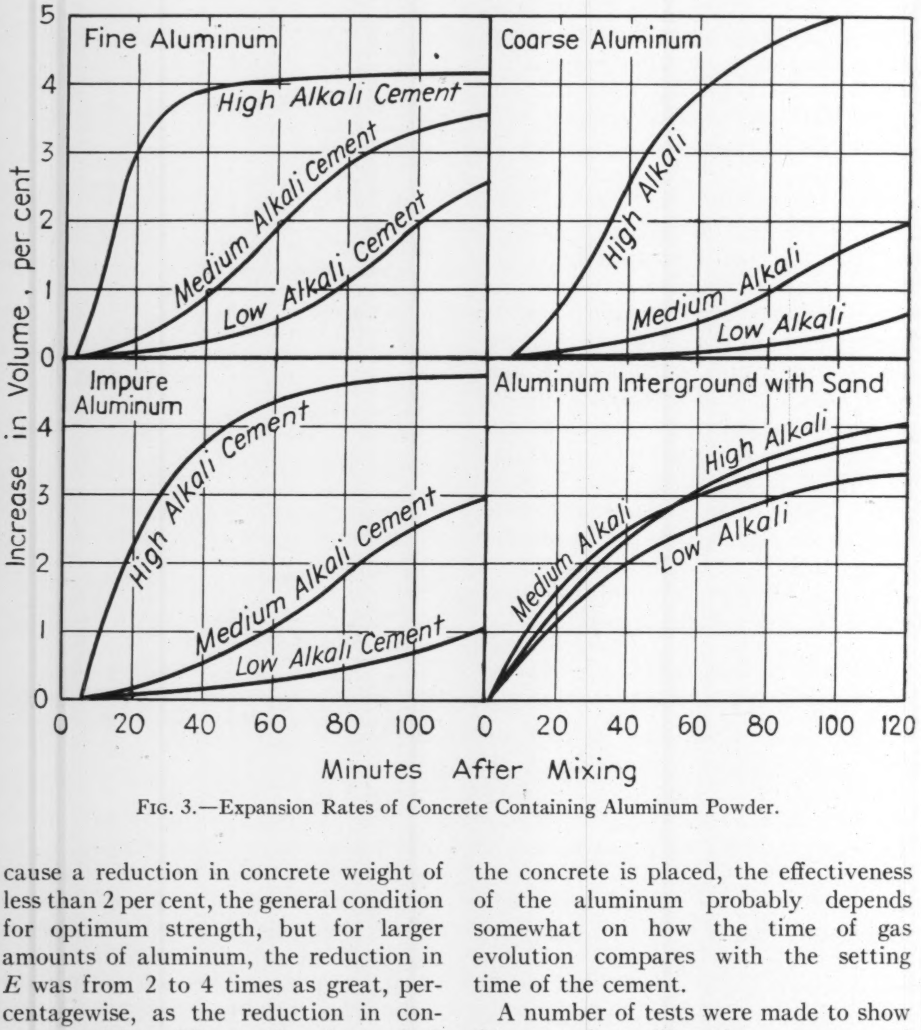


FIG. 3.—Expansion Rates of Concrete Containing Aluminum Powder.

for making the tests consisted of a 3 by 6-in. metal cylinder with an overflow pipe near the top. The cylinder was filled to within a ½ in. of the overflow with concrete, the remainder of the space to the overflow being filled with water. A metal disk of 2.5-in. diameter was partially submerged in the water to magnify the tendency of the surface to rise, and a wetting agent was added to the surface water. A steady drip of water from the overflow then occurred due to any expansion of the concrete, and this drip was collected and measured.

In Fig. 3 are shown the rates of expansion of 12 concretes, comprising 3 cements differing as to alkali content, and 4 grades of aluminum powder. The expansion tests were made with a mix of 1:4 by weight and an aluminum content of 0.014 per cent. The 4 grades of aluminum powder were as follows:

(*a*) Commercial powder, finest grade, 420 mesh, polished with tallow, relatively pure,

(*b*) Commercial powder, coarse grade, 160 mesh, polished with tallow, relatively pure,

(*c*) Commercial powder, 200 mesh, from paper-backed scrap and therefore impure, probably containing some lead and tin, and

(*d*) The 420 mesh powder of (*a*), preground with silica sand in proportions 1:50 by weight to about 1500 sq.cm. per g.

Referring to Fig. 3, it is evident that the expansion rate of the concrete is related to the alkali content of the cement, *except* when the aluminum is preground with sand. The beginning of rapid expansion is delayed many minutes when the alkali content of the cement is reduced. The actual alkali contents of the three cements in Fig. 3 were 1.2, 0.7, and 0.2, respectively. It is possible that the dependence of expansion rate on alkali content of cement is due to the trace of tallow remaining in the aluminum powder from the polishing operation in its preparation. This seems to be the only ready explanation for the absence of any important effect of alkali content of cement when the aluminum was preground with sand, whereby the traces of tallow probably were removed.

The impure aluminum was included in the tests mainly because it is obtainable even in time of war. Being impure, its reaction rate could be expected to be different, but its rate was found to be just as predicted for pure aluminum of comparable fineness. Tests are now being made of the relative effects of the impure aluminum on the other concrete properties.

The total expansion observed in the concretes was considerably less than the theoretical amount. If all of the aluminum reacted, if no gas escaped, and if the gas were under atmospheric pressure, the total expansion under the conditions in Fig. 3 should have been about 7.7 per cent. However, some gas undoubtedly does escape and the concrete restrains the expansion so that the gas is under some pressure. In predicting the approximate expansion likely to occur, a convenient procedure is to multiply by 1800 the percentage of aluminum by weight of concrete. Thus, 0.014 per cent of aluminum in a 1:6 mix with a water-cement ratio of 0.60 would be 0.0018 per cent by weight of concrete, and the expected expansion would be (0.0018) (1800) or 3.2 per cent. This expression takes account of the richness of mix and incompleteness of expansion, but it does not take account of any unusual restraint or confinement of the concrete that would tend to reduce the expansion.

TENTATIVE CONCLUSIONS

The tests reported herein are not comprehensive enough to permit drawing of final conclusions, but the following tentative conclusions seem to be warranted:

1. A large improvement in resistance to freezing and thawing with calcium chloride can be obtained by adding a sufficient quantity of finely-ground aluminum to cause a reduction in the concrete weight of several per cent; if the aluminum is interground with the cement, up to 0.020 per cent of aluminum is indicated, and if the aluminum is simply admixed, up to 0.014 per cent is indicated.

2. Bond strength to reinforcing steel is improved markedly by the addition of from 0.005 to 0.014 per cent of finely ground aluminum to the concrete mix.

3. Flexural strength of concrete can be improved appreciably by the addition of from 0.005 to 0.014 per cent of aluminum powder, even though the attendant compressive strength may be decreased.

4. The compressive strength of all but harsh mixes and confined concrete is reduced by the addition of aluminum powder to the mix.

5. Optimum strength usually is obtained with about 0.007 per cent of aluminum powder by weight of cement, but optimum resistance to freezing and thawing with calcium chloride is obtained with two to four times as much aluminum.

DISCUSSION

Mr. Benjamin Wilk.[1]—I should like to ask whether it would be desirable to add the aluminum to the water or to the aggregate?

Mr. Roy W. Carlson.[2]—It is usually added to the aggregate, although it probably makes little difference how it is added if it is preground with sand, one part of aluminum to 50 or 100 parts of sand. We have never tried adding it to the water. We have always added it to the cement or the aggregate.

Mr. R. E. Davis.[3]—We have found that better dispersion is obtained by adding the aluminum to the aggregate. While ours is on a small laboratory scale, I believe that if the additions were being made in a mixer it would be best done as Mr. Carlson suggested, namely, by first pregrinding it for a very short time with dry sand. That will insure getting good dispersion in the mix.

Mr. F. C. Lang.[4]—I was a little surprised at the increase in bond strength. Tests made in our laboratory on concrete in which vinsol resin or natural cement - portland cement blends were used showed a decreased bond strength. I wondered whether the increase in bond strength was caused by compression, due to the formation of gas.

Mr. Carlson.—The increase in bond strength in this case seems to be the elimination of the bleeding of the water and the contact with the bar on all sides. In the same series of tests we made some tests of the introduction of vinsol resin, and there was some reduction in bond strength with the introduction of vinsol resin. We did not include those tests because we did not think there were a sufficient number to include them.

Mr. H. F. Gonnerman.[5]—We have made a good many tests of concrete containing powdered aluminum and are preparing a paper on this work. Although we have covered many of the phases Mr. Carlson has reported upon we have never been able to obtain an increase in compressive strength of plastic concrete in standard 6 by 12-in. cylinders unless the expansion of the concrete was restrained during the early hardening period. By using suitable quantities of aluminum powder we have found improvement in bond resistance and in resistance to freezing and thawing, as Mr. Carlson has pointed out.

I should like to ask what explanation Mr. Carlson has for the results he reports that when the aluminum was ground with the cement it was not quite as effective as when it was added to the mix?

Mr. Carlson.—For a given amount of gas generation, the aluminum appears to be more effective when ground with the cement. But it is necessary to use two or three times as much aluminum ground with the cement for a desired amount of gas generation. The aluminum appears to react somewhat with the cement before use.

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[1] General Manager, Standard Building Products Co. Detroit, Mich.

[2] Associate Professor of Civil Engineering, Massachusetts Institute of Technology, Cambridge, Mass.

[3] Professor of Civil Engineering, and Director, Engineering Materials Laboratory, University of California, Berkeley, Calif.

[4] Engineer of Materials and Research, Highway Department Laboratory, Minnesota Highway Dept., Minneapolis, Minn.

[5] Manager, Research Laboratory, Portland Cement Assn., Chicago, Ill.

MR. WILLIAM J. MAYER.[6]—Have any tests been made to determine the thermal conductivity of concrete with aluminum?

MR. CARLSON.—No, we have made none. We would not expect it to have much effect, because the percentage by weight of the cement is only 0.007 per cent, and by weight of the concrete roughly 0.0007 per cent.

MR. NICOLAAS T. F. STADTFELD.[7]—I was quite surprised in the author's statement that portland cement containing only 0.2 per cent of total alkalis would be affected by the addition of aluminum. The reason I mention this is that I know of one cement, low in alkalis, which could not be used in aerocrete as there was not enough reaction with aluminum to form the necessary hydrogen.

The problem you investigated is, to some extent, different from the use of aerocrete; still I should like to know whether the effect of alkali content was noticeable when it differed as much as from 0.2 per cent to 1.0 per cent?

MR. CARLSON.—When the aluminum was not preground with sand, there was practically no effect. We believe the polished aluminum to be coated with a trace of tallow, and it takes a certain amount of alkali to break that down. If it is preground with sand so as to remove the tallow, it does not seem to make any difference; the rates are almost identical.

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[6] Manager, Engineering Service Dept., A. M. Byers Co., Pittsburgh, Pa.

[7] Division Engineer, New York City, Board of Water Supply

THE EFFECT OF BRICK ABSORPTION CHARACTERISTICS UPON MORTAR PROPERTIES

By F. O. Anderegg[1]

Synopsis

Absorption characteristics have been determined on several types of brick taken from different parts of the kiln. The abstraction of moisture by these bricks from lime mortar after contact lasting 1, 10, and 100 min. has been measured, and the setting times have been taken. The moisture gradients in lime mortars in contact with these bricks were also observed, and the effect of high initial absorption on mortar properties caused by the congealing of the mortar surface are described. Bond and joint-mortar compressive strengths, obtained with a 1:2:9 mortar at 28 days in contact with several types of brick, are plotted against 1-min. absorptions. The result on mortar strengths of wetting 3 of the more highly absorbent bricks is also shown graphically. Some observations on the effect of brick absorption on mortar durability are given. Strengths are recorded for joint mortars varying in composition from straight lime to straight portland cement for ages up to 1 yr. Their ratios to metal molded cube strengths are also given permitting one to make an estimate of mortar strengths in the wall. The problem of mortar durability is discussed in light of the British rule for choice of a mortar, and the need for more study of the deformability of masonry assemblages is emphasized.

That the strength of mortar in masonry joints is affected by the absorption behavior of the units in contact therewith, has been reported with respect to stiff-mud bricks.[2,3] This study has now been extended to include other mortar properties and other types of brick.

Brick Properties

The types of brick included in these experiments are listed in Table I, together with some of their properties.

As far as possible, bricks were selected from the top and bottom courses of the kiln and from one intermediate section. The values given under absorption are averages, but in every case a considerable amount of scatter was encountered. Some bricks from the middle of a kiln would be similar to the average from the top, while others would be like typical bricks taken from lower courses. Indeed, as Stull and Johnson[4] have pointed out, considerable variations in pore structure are often encountered in different parts of the same brick.

In selecting bricks for experimentation, the 1-min. absorption of all the stiff-mud bricks was determined at the brick yard and those bricks ranging from 1 to 5 g. were sorted out as brick *A*. For bricks *B* and *C* the limits were 16 to 24 g. and 45 to 55 g., respectively. The other types of brick were shipped some distance and it was necessary to use those available. In each case, however, they were sorted according to the 1-min. absorption and the values given in the table represent the range within ±20 per cent. Comparisons of various properties were also made with 10-min. or 24-hr. immersion values, but in all cases the data, when plotted against 1-min. absorptions, deviated less from smooth lines than when the longer-period absorption values were used.

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[1] Consulting Specialist on Building Materials, Newark, Ohio.

[2] F. O. Anderegg, "Watertight Brick Masonry," *Architectural Record*, p. 208 (1931).

[3] F. O. Anderegg, "Some Properties of Mortars in Masonry," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 1130 (1940).

[4] Ray T. Stull and Paul V. Johnson, "Some Properties of the Pore System in Bricks and Their Relation to Frost Action," *Journal of Research*, Nat. Bureau Standards, Vol. 25, p. 711 (1940); "Relation Between Air and Water Permeabilities of Building Brick," *Bulletin*, Am. Ceramic Soc., Vol. 20, p. 443 (1941).

TABLE I.

Brick Type	Material	Source	Absorption 1 min., g.[a]	Absorption 10 min., per cent	Absorption 24 hr., per cent	Surface Condition
A Stiff-mud, side-cut	Shale	Central Ohio	3	0.5	1.4	rough
B			20	4.7	6.5	
C			50	8.0	13.0	
D Soft-mud, water struck	Clay	New Hampshire	3	0.4	1.1	fairly smooth
E			10	3.0	5.5	
F			50	10.0	12.0	
G Soft-mud, sand struck	Clay	Central Penna.	70	6.5	12.0	fairly smooth
H Soft-mud, sand struck	Clay	Hudson Valley	45	7.5	9.0	fairly smooth
H′ Same as *H* except for a whitish flat face			60	10.0	13.0	
M Dry-press	Shale	N.W. Penna.	80	6.0	7.0	very smooth
S Dry-press nonceramic	Sand	New York City	120	8.0	12.0	very smooth

[a] The 1-min. absorption was determined by placing a brick flat in ¼ in. of water for exactly 60 sec. The others involved complete immersion.

It was hoped to be able to tie in the shape of the absorption curve, when plotted against time, with the pore structure; but too little is known about the latter to permit more than broad generalizations. The very rapid initial rise of water in the dry-press bricks indicates the presence of relatively large pores. On the other hand, the very slow absorption of the hard-burned bricks might be interpreted as due to the pores being rather fine. However, Stull and Johnson[4] obtained indications that such pores were few in number and relatively large. Here is a nearly virgin field needing experimental study.

The smoothness or roughness of the brick surface should receive consideration. In the first place, it is difficult for a mortar to come completely in contact with a rough surface brick, so that suction on the mortar would be reduced. The bond would be lowered, while leakage would increase. On the other hand, where contact is complete, a rough surface will give a mechanical keying which is desirable. Such contact is most easily obtained with bricks of lower absorption rates, more or less compensating in the bond strength for a higher water-cement ratio in the mortar.

MORTARS

For a study of the effect of the different types of absorption on the water content of a mortar, it is important to select a mortar of constant properties. Such a mortar was obtained by allowing lime to soak for at least two weeks, followed by prolonged mechanical mixing with the required amount of sand. In this way the clumps of lime solids were broken down, so that additional handling would cause little change. Two limes were used, one, a high-calcium, high-yield quicklime *Q*, and the other, an Ohio dolomitic hydrated lime *R*, in which the magnesia was only slightly hydrated. They were mixed 1:3 with local sand[5] (50 parts lime solids to 240 parts dry sand by weight). With a water-solids weight-ratio close to 0.305, the consistency was maintained to give a flow of 110 per cent. The mortars made from the two limes so prepared behaved almost alike.

For strength tests, a 1:2:9 mortar was used, or 94:100:720 parts by weight.[5] The cement was a blend of four standard brands, meeting the requirements for Type 1 of the A.S.T.M. Standard Specifications for Portland Cement (C 150 – 41).[6] The lime was a pressure-hydrated dolomitic lime, with strength and workability very similar to the average values previously obtained with 20 different brands of lime. The sand and methods used have been previously described.[3] Some of the properties of these mortars are given in Table II.

TABLE II.—PROPERTIES OF MORTARS.

Mix	Flow, per cent	Penetration, mm.[a]	Splash, in.[b]	Moisture, *W/S*, per cent	Air Volume, per cent	Unit Weight, lb. per cu. ft.	Volume Paste / Absolute Volume Sand	Water Retention, per cent	Compressive Strength at 28 days, psi.	
									Cube	Joint Mortar[e]
0:1:3..	110	40 ± 4	6.6 ± 2	30.5	1.0 to 1.5	114 ± 2	1.06*Q* 1.00*R*	85*Q* 87*R*	70*Q*[c] 80*R*[c]	180*Q* 200*R*
1:2:9..	110	25 ± 3	6.6 ± 2	22.4	0.4 to 1.0	124 ± 2	1.11*Q* 1.08*R*	80	750[d]	1580

[a] Penetration of a cylinder of 42-mm. diameter, weighing 700 g. into mortar held in a cup 9 cm. deep and with an internal diameter of 10 cm.

[b] Splash is the average diameter of mortar which has fallen 15 cm. onto a plate. The flow table mold is placed on a wet glass plate, and after filling, the plate was removed rapidly in a horizontal direction.

[c] Metal-molded 1-in. cubes stored in the laboratory air.

[d] Metal-molded 2-in. cubes stored in water.

[e] Taken from joints between stiff-mud bricks having a 20-g. 1-min. absorption.

ABSORPTION OF MOISTURE FROM MORTAR

When mortar was placed in contact with any of the brick used in this investigation some moisture was immediately removed. The rate of removal and the total amount abstracted had a considerable effect on the mortar properties. A study has been made of the abstraction of moisture from mortar by the different bricks, using the lime mortars. Two air-dried bricks were weighed and 500 g. of mortar was placed on one brick and spread to a uniform bed in 30 sec. The second brick was then placed on top and pushed down to produce a joint ½ in. thick. The mortar which was squeezed out was returned to the joint with an upward cutting motion of the trowel. The pair was then inverted. After 1, 10, or 100-min. contact, the top brick was detached and brushed free of all mortar and weighed. This was repeated with the other brick. The results are plotted in Fig. 1.

Some of the same mortar was placed on similar bricks in a metal ring ½ in. thick and the setting time was noted with the heavier Gillmore needle at the center of the ring.[7] These setting times are plotted as squares in Fig. 1. Similar setting times were taken with the 1:2:9 mortar. They are also plotted in Fig. 1, although abstractions were run on lime mortars only.

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[5] Report of Committee C-7 on Lime, Evaluating Cubic Foot Quantities of Mortar Materials, *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 277 (1941).

[6] 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 1.

[7] L. A. Wagner's Method, personal communication.

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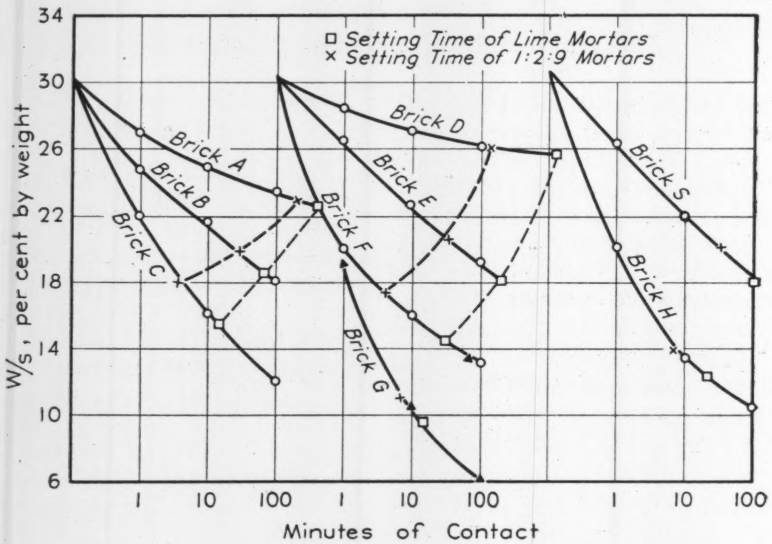

FIG. 1.—Abstraction of Moisture from Lime Mortar by Different Bricks.

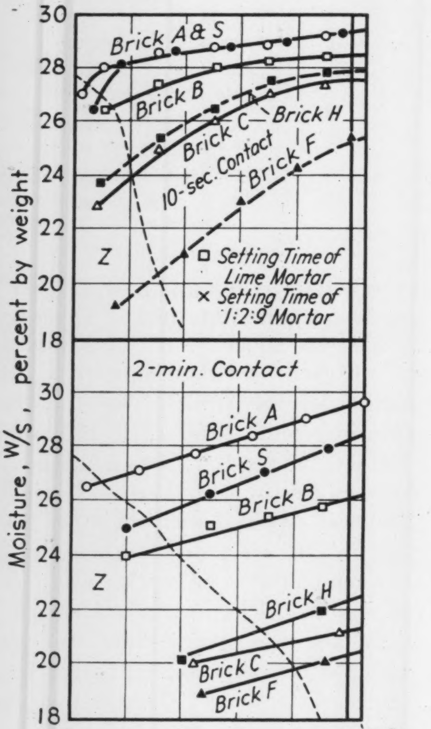

FIG. 2.—Moisture Gradient in Lime Mortar in Contact with Different Bricks.

The areas at the lower left corner marked Z indicate the approximate thickness of the congealed layer.

It is surprising to note that the type of brick having the highest initial rate of absorption of water removed but a very small amount of moisture from these lime mortars. Only the two very hard-burned bricks, *A* and *D*, abstracted less. Another surprising result was that before the mortar set up, a great deal more total moisture had to be abstracted by the softest burned brick in a given series than by the hardest burned member. This held for both the lime mortars and the 1:2:9 mortar, suggesting the existence of marked differences in moisture gradient within the mortars in contact with the different bricks. For bricks taken from the same kiln, it was noted that the product of the 1-min. absorption in grams and the setting time in minutes, was a constant.

MOISTURE GRADIENT AND CONGEALED LAYER

To obtain information about the moisture gradient within the mortar, lime mortar was placed in contact with a given brick for definite periods of time. The mortar was then gently removed from the brick. The layer left clinging to the brick was sampled as well as six successive 1-mm. layers taken from the detached mortar. Then the moisture content of each layer was determined. The results obtained after contact lasting 10 sec. and 2 min., are given in Fig. 2.

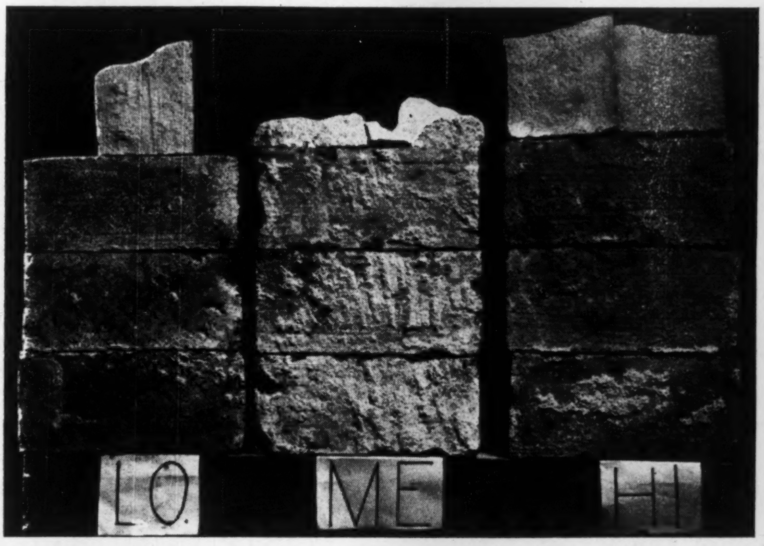

FIG. 3.—Beams Made from 1:2:9 Mortar and Brick of Low, Medium, and High Absorption After Breaking.

The mortar clung tightly to the bricks of medium absorption

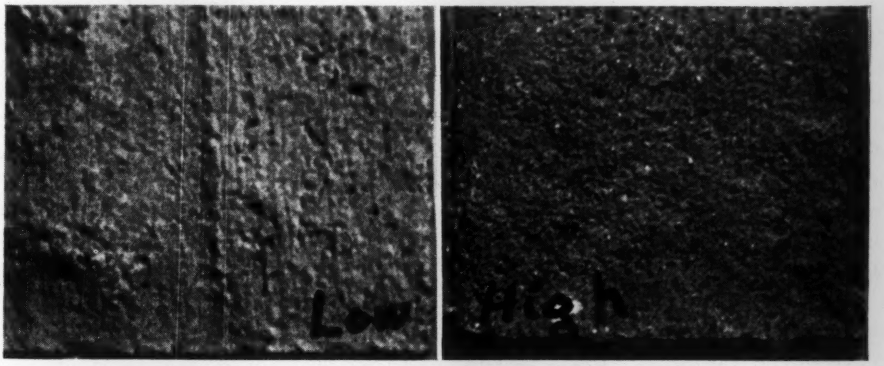

FIG. 4.—Enlarged Photographs of the Mortar Surface Shown in Fig. 3.

The mortar from the hardest burned bricks bears a faithful imprint of their surfaces. The surface of the mortar from the highly absorptive bricks had been congealed too rapidly to permit making complete contact. Note the depressions resulting. Also note the difference in whiteness, which reflects the difference in water-cement ratio.

It is interesting to note that among the *more* absorptive bricks, most moisture was removed by some of the bricks having lowest initial absorption for water, confirming the results obtained in the first experiment. One reason for this anomalous behavior was disclosed by these experiments. The higher the initial absorption rate, the greater was the apparent tendency to form a highly congealed layer at the surface of the mortar. The rest of the mortar could be readily peeled from the congealed layer. Moisture is apparently removed so rapidly from the surface of the mortar by some of the bricks of high initial suction capacity that a condensed layer is formed having a permeability varying inversely with the initial suction. The thickness of this layer is indicated in Fig. 2 by the shaded area on the left marked Z. In this way a steep moisture gradient was built up between the surface layer and the interior of the mortar. The effect of this phenomenon on the setting time and on some of the other mortar properties was often quite great.

AREA OF CONTACT

Another effect of the initial high suction rate was the prevention of the mortar from flowing out over, and making continuous contact with, the surface of the brick. The effect of this on the surface of the mortar in contact with a high absorption brick is shown in Fig. 3. More detail is given in Fig. 4, which is an enlargement of the mortar surfaces removed from both high and low absorption bricks. The surface of the latter is a faithful imprint of the brick surface, while the mortar surface from the higher absorption brick is characterized by hills and valleys. The rapid congealing of the mortar has left a network of capillaries between brick and mortar. This effect has been noted previously,[2] where its presence was connected with wall leakage. It was there associated with poor water retention in the mortar, while the effects here exhibited were obtained with mortars having not only excellent water retention but also other properties contributing to the best workability. It is evident that the units play a part in the watertightness of the wall.

BOND STRENGTH AND JOINT-MORTAR COMPRESSIVE STRENGTH

A considerable number of 2-brick beams have been made up with bricks of different types and absorption rates. They were broken at 28 days with cantilever loading and specimens cut from the joint-mortar were crushed as previously described.[3] The results obtained have been plotted in Fig. 5, against the 1-min. absorption as abscissas. These values were confirmed by applying a single batch of mortar to 6 different types of bricks. The bond

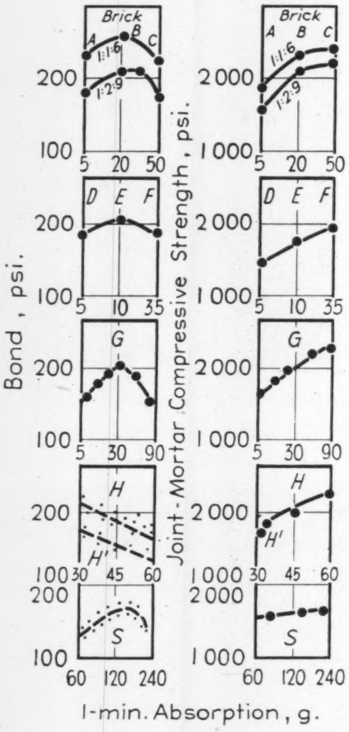

FIG. 5.—The Effect of Absorption Rate of Various Bricks upon Bond Strength and upon Joint-Mortar Strength of a 1:2:9 Mortar.

strengths were obtained by averaging the results from 3 to 6 beams. Their average deviation ran about 5 per cent. The compression data were obtained from 10 to 20 specimens, and the average deviation ran about 15 per cent.

For most of the different types of bricks the bond strengths increased with the brick absorption to a maximum, beyond which they decreased. The first increase was due to reduced water-cement ratio within the mortar, while the decline resulted from mortar surface congealing and from the consequent poor contact. The decreasing bond strengths of bricks *H* and *H′* indicate that they have absorption rates above the optimum and should be wetted, as is recommended by the producers.

The compressive strength of mortar taken from the joints increased with the absorption rate, not only to the point where bond strength was a maximum, but sometimes even beyond that. This was doubtless caused by a lowered water-cement ratio. However, with mortars of higher portland cement content, strength decreases in mortar in contact with highly absorbent bricks have been occasionally noted, no doubt due to the fact that insufficient moisture was left in the mortar to cure the cement properly.

High-lime mortars in contact with the nonceramic bricks *S* behaved in a peculiar way. They set more slowly than they did when in contact with any other of the bricks studied, except those which were very hard burned. As was stated above, the formation of a very dense congealed layer next to the bricks resisted the transmission of moisture from the center of the mortar joint. This resulted in comparatively low compressive strengths. A similar effect has been noted with certain highly absorptive ceramic bricks.

The continuing rise of compressive strength to a maximum as absorption increases may in part be attributed to the effect of the numerous tiny capillaries in bricks *C*, *F*, and *G*, reported by Stull and Johnson[4] for similiar bricks. In spite of the formation of the congealed layer, there seems often to be sufficient suction force to continue the abstraction of moisture over a considerable period of time.

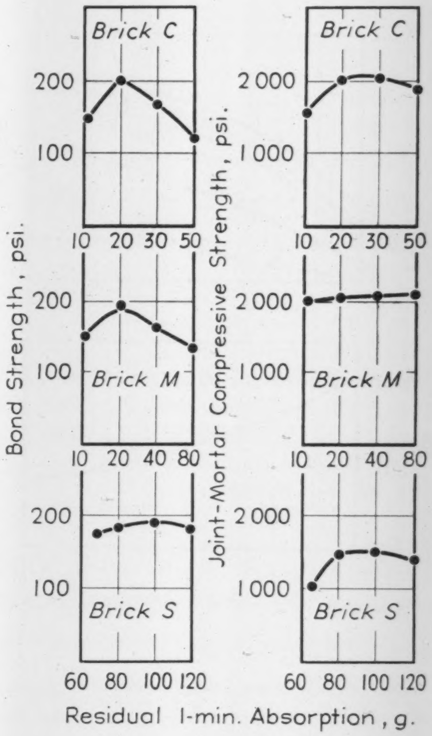

FIG. 6.—The Effect of Wetting Absorptive Brick upon Bond Strength and upon Joint-Mortar Strength of a 1:2:9 Mortar.

EFFECT OF WETTING THE UNITS

Three of the more absorbent bricks, *C*, *M*, and *S*, each received applications of water to that surface on which 1:2:9 mortar was to be applied, sufficient to reduce the 1-min. absorption to the abscissa values given in Fig. 6. Beams were made from these bricks and also from similiar dry bricks. Bond and compressive strengths are recorded in Fig. 6. The values for the dry bricks are plotted on the right-hand side of the figures. As water was added to ceramic units *C* and *M*, the bond strength increased to a maximum and then declined. The increase was of the order of 70 per cent. Nonceramic unit *S* behaved in a peculiar way which is receiving more investigation.

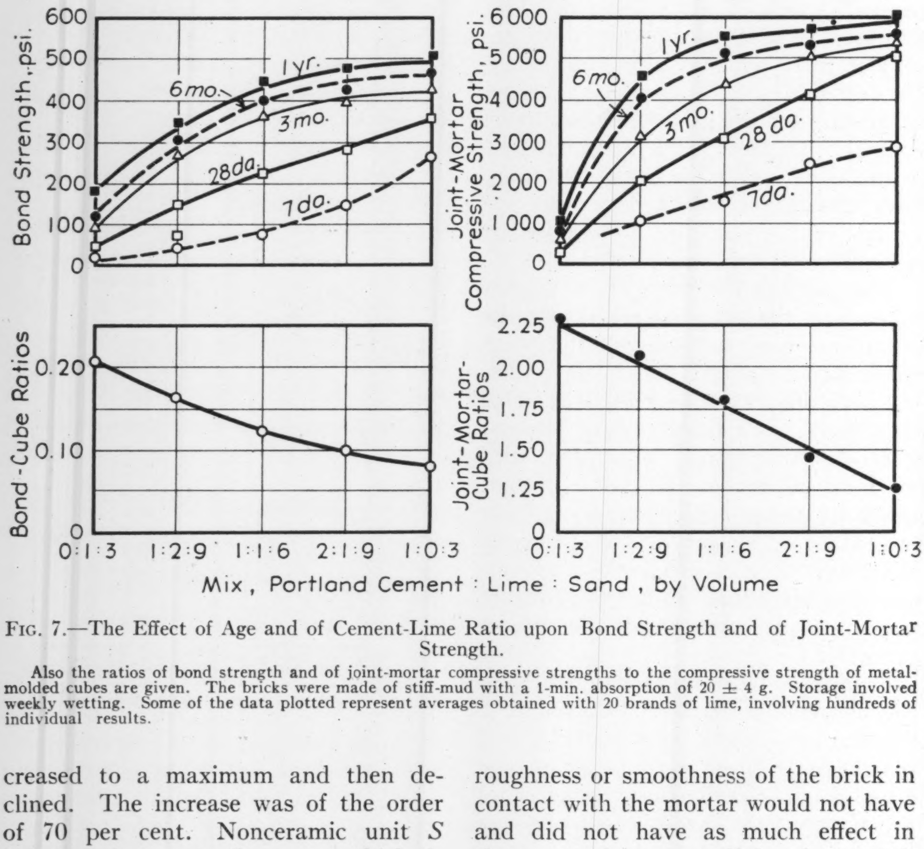

FIG. 7.—The Effect of Age and of Cement-Lime Ratio upon Bond Strength and of Joint-Mortar Strength.

Also the ratios of bond strength and of joint-mortar compressive strengths to the compressive strength of metal-molded cubes are given. The bricks were made of stiff-mud with a 1-min. absorption of 20 ± 4 g. Storage involved weekly wetting. Some of the data plotted represent averages obtained with 20 brands of lime, involving hundreds of individual results.

While most brick masons appreciate the importance of wetting the highly absorbent bricks, in the rush of erection this detail is often neglected. More care should be used in wetting, or such highly absorbent bricks should not be selected, where bond strength and water-tight walls are of importance.

EFFECT OF SURFACE TEXTURE

The method of testing for bond strength depends upon stress in the extreme fiber, since the beams were broken by cantilever loading. The roughness or smoothness of the brick in contact with the mortar would not have and did not have as much effect in these experiments as if bond strength had been determined by shearing tests.

EFFECT OF BRICK ABSORPTION ON DURABILITY

The results on durability previously reported[3] have been extended to include mortars made from 10 different brands of lime, in different ratios with portland cement, fabricated into 2-brick beams with bricks *A*, *B*, and *C*. After 28 days storage in the laboratory half were placed in a pan on the roof and the rest were half-buried in the ground. In all cases, with one exception, which was a brand of incompletely-hydrated dolomitic lime, the 1:2:9 mortars failed during the first winter when in contact with hard-burned brick *A*. Most of the mortars in contact with the more absorbent bricks, *B* or *C*, were in excellent condition at the end of the first winter. However, a few 1:1:6 mortars made from high-yield, high-calcium lime putties failed during the first winter when in contact with the hardest burned bricks. After the second winter of exposure, little apparent change had occurred with the assemblages containing the bricks of medium absorption. About half of the pairs containing the high absorption bricks were split apart, presumably by the freezing of water in the capillaries lying between the bricks and the mortar. The 1:2:9 dolomitic lime mortar, which had been placed between hardest burned bricks and which was mentioned in the preceding paragraph, still appeared to be in good condition. This study is being continued.

PREDICTING JOINT-MORTAR AND BOND STRENGTHS FROM CUBE STRENGTHS

Strength values obtained with about 20 brands of lime, in various ratios with portland cement, have been averaged and may be found in Fig. 7. The results include those previously reported[3] and a great many more obtained since. The data at 6 months and at 1 yr. were secured with 8 brands of lime.

In order to take advantage of the convenient cube test for mortar strength, data are also given in Fig. 7, showing the ratio between joint mortar strengths as determined by those methods and the strengths obtained with wet-stored, metal-molded cubes. This relation seems to hold reasonably well for ceramic bricks having a 1-min. absorption range between about 10 and 100 g. With units of lower absorption the ratio approaches unity. The ratios of bond strengths to cube strengths are also included in Fig. 7. In practise the application of the ratios is more uncertain because of the deleterious effect on the bond of the discontinuities, resulting from incomplete joint filling and, especially, from inadequate tooling of the joints.

These joint-mortar compressive strengths are so high as to raise the question of choice of mortar for a given brick. The British have an excellent rule,[8] which seems to be borne out in practise. This rule might be stated: "Choose mortar having properties similar to, but somewhat weaker than, those of the units. The mortar should have some of the functions of a safety valve, and safety valves should not be tied down."

This rule has been applied in considering the relative resistance of units and mortar to efflorescent disintegration, which is apparently the chief hazard to durability of masonry walls in Britain. It has also been suggested as being the chief cause of the disintegration in this country of limestone,[9] concrete[10] and brick masonry.[2] The compressive strength of masonry mortar, so far as load-carrying ability is concerned, does not generally cause much worry. The *durability* of the mortar would seem to be more important. However, for lack of a better criterion, durability has often been regarded as synonymous with mortar strength. While strength is a factor in durability, the pore system is much more important since disintegration will not occur where there is lack of opportunity for corrosive solutions to penetrate a system.

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[8] F. Fitzmaurice, "Principles of Modern Building, Vol. I," pp. 245–247, H. M. Stationery Office, London (1939).

[9] F. O. Anderegg, H. C. Peffer, P. R. Judy and Lee Huber, "Indiana Limestone I, Efflorescence and Staining," *Bulletin No. 33*, Purdue University Engineering Experiment Station (1928).

[10] F. O. Anderegg, "The Mechanism of Corrosion of Portland Cement Concrete with Special Reference to the Role of Crystal Pressure," *Proceedings*, Am. Concrete Inst., Vol. 25, p. 332 (1929).

The whole philosophy of the masonry wall needs careful review and much more experimentation is required on masonry assembledges to permit proper evaluation of the importance of the various mortar properties.

Durability against efflorescent disintegration or against freezing is suggested to be more important than mere strength; but it should be balanced against the cushioning action, apparently desirable in the mortar, to take up the movements of the walls which are constantly going on. The British rule might be advantageously applied to mortar deformability, as well as to the resistance to efflorescent action.

Experiments in hand involving measurements or the modulus of elasticity of joint mortars indicate attainment of rather high values, about a thousand times the compressive strengths given in Fig. 7. These observations indicate high mortar rigidity and the question may fairly be raised, "Have we not been overemphasizing strength in mortar?" The answer can be obtained only by careful experimentation. The study of the deformability of masonry assembledges has been inaugurated in this laboratory.

SUMMARY

Before the mortar at the joint surface sets up, much more moisture is abstracted from a lime mortar by a highly absorbent brick than by bricks of low absorption. A steeper moisture gradient also occurs. The effect of the rapid absorption is to produce a congealing of the surface layer. This congealed layer seems to offer more resistance to moisture movement. Another result of this action is to prevent the mortar from making intimate contact with the brick surface, so that bond strengths are poor. A network of capillaries is left through which rain may penetrate. If such brickwork becomes saturated and frozen, the mortar is likely to be separated bodily from the bricks. Bricks of high absorption should be wetted before laying or not used where bond strength or watertightness is important.

Bricks of very low absorption remove moisture so slowly that their laying becomes more difficult. The setting time of the mortar is considerably increased. Both bond and joint-mortar compressive strengths are low, while mortar durability is adversely affected.

Bricks of intermediate absorption are easiest to lay. Mortars in contact with them have setting times which are convenient for the mason. The bond strengths often run double, those obtained with bricks having very high or very low absorption, while the joint mortar strength is close to the maximum. The durability of the joint is also greatest with brick of medium absorption.

Bond and joint-mortar compressive strengths are given for mortars ranging from straight lime to straight portland cement. Surprisingly high results are obtained at 6 months and 1 yr. for 1:1:6 mortars, and the question arises whether such mortars have too great rigidity. The British rule for choice of mortars, where efflorescent action is the chief consideration, would seem also logical to use where deformability is concerned. The problem of securing proper balance between rigidity and durability needs more experimental study.

Acknowledgment:

Grateful acknowledgment is due to William Uffner for able assistance in obtaining the results reported here.

DISCUSSION

Mr. Howard R. Staley.[1]—Mr. Anderegg's paper has raised a point which has considerable bearing upon the successful use of mortar in masonry work. This is the function of the mixing water in the mortar. Water is the only liquidizing factor and is also the means by which cohesiveness is imparted to the dry materials.

The common conception is that the mixing water has performed the function for which it is intended if the mortar is placeable in the wall. It is my conception that the water has a function to perform during the placement of the unit and for some period after the unit is put in place. Water is the vehicle which allows of placement of the mortar, and it is the vehicle by which some benefit in density of the mortar can accrue if some of it is held in the mortar due to the water retentivity of the cementitious materials. The addition of the wall load during erection of the wall causes the mortar to flow plastically after some water has been lost to the unit thus densifying the mortar and also enabling it to come into more intimate contact with the unit. One of the reasons for the disintegration of parapet walls is due to the fact that the wall load is small at the time of erection and later when the wall is in service.

If a rapid-setting mortar or one of low water retentivity is used with a highly absorptive brick, the mortar will attain a state of rigidity almost immediately, due to loss of water alone. Thus the mortar becomes too rigid to flow plastically, and it has to bear the increasing wall load without being benefited by consolidation. This process leaves the mortar most porous at the brick line, where the greatest water loss has occurred, and leaves spaces through which water may permeate. It has been fairly well established that practically all of the visible leakage of water travels through the wall at the junction of the brick and the mortar.

At the other extreme, if a mortar of low water retentivity is used with a brick of very low absorption, then the water will not be lost to the unit and some benefit may accrue in density as well as extent of bond. From a practical standpoint this is not successful, as the mortar will "bleed" its water to the face of the wall leaving a smeared surface which is very difficult to clean. If such a mortar is slow setting then more benefit will be obtained due to consolidation than if the mortar is rapid setting, for as the water bleeds to the surface the fluidity of the mortar allows the masonry to consolidate due to increasing wall load. Some "floating" is apt to occur and the difficulties of rapid erection of the wall increase. The practical difficulties of using such a combination of materials should be emphasized as the cost of the resulting masonry would be high.

Mr. Anderegg's paper brings out some of these points. His results also emphasize the fact that the absorption characteristics of the unit have quite as much to do with the successful use of masonry materials as the characteristics of the mortar. There are many factors, such as reported by Mr. Anderegg, that require a great deal more work. It is the opinion of the writer that more members of the Society and of Committee C-12 on Mortars for Unit Masonry specifically should be making some contribution to this work.

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[1] Assistant Professor of Building Construction, In Charge of Building Materials Research Laboratory, Massachusetts Institute of Technology, Cambridge, Mass.

MR. A. T. MALMED.[2]—I should like to ask Mr. Staley and Mr. Anderegg a question on this point. Some years ago Mr. Voss brought out the theory of a bond layer. He contended that by using mortar high in lime a bond layer of lime is created and that it is this bond layer of lime that made it possible for the mortar to cling tightly to the surface of the brick.

Now Mr. Anderegg evidently does not think much of this bond layer theory because he writes in his paper: "Before the mortar at the joint surface sets up, much more moisture is abstracted from a lime mortar by a highly absorbent brick than by bricks of low absorption. A steeper moisture gradient also occurs. The effect of the rapid absorption is to produce a congealing of the surface layer. This congealed layer seems to offer more resistance to moisture movement. Another result of this action is to prevent the mortar from making intimate contact with the brick surface so that bond strengths are poor. A network of capillaries is left through which the rain may penetrate. If such brickwork becomes saturated and frozen, the mortar is likely to be separated bodily from the bricks. Bricks of high absorption should be wetted before laying or not used where bond strength or watertightness is important."

Now I should like Mr. Anderegg and Mr. Staley to enlighten me on this difference of opinion between them and Mr. Voss concerning this bond layer. As I understand Mr. Voss's theory of the bond layer, its importance is its supposed ability to cling to the surface of the brick. Now according to Mr. Anderegg's paper, this supposed bond layer acts the direct opposite. He contends that it is practically dead matter and that under freezing and thawing action this bond layer actually separates from the surface of the brick. It seems to me that he and Mr. Voss do not agree on the function of the bond layer.

MR. STALEY.—As I see it, these opinions are not divergent.

MR. MALMED.—Does Mr. Voss's assistant, Mr. Staley, believe that there is such a thing as a bond layer?

MR. STALEY.—Essentially, yes. Mr. Anderegg has shown that there is a congealed layer formed in some instances, at the brick surface, due to the rapid withdrawal of water. That congealed layer might become more impervious to water from the mortar due to the fact that lime or at least cementitious material which is in suspension or in solution would be carried to the surface of the brick by the suction of the unit. The porosity of the mortar might be increased because of the rapid withdrawal of water and the rapid attainment of rigidity due to loss of water alone. The permeability to water traveling parallel with the layer would probably be increased. The bond layer would be the result of the carbonation of the thin layer of cementitious materials at the brick surface.

MR. MALMED.—After comparing the explanations of the bond layer made by Mr. Anderegg and Mr. Voss, as a manufacturer of a masonry cement we certainly are confused as to whether this bond layer is important or whether it is a definite hindrance.

MR. STALEY.—I prefer to call it just bond. Such a layer, when carbonated, would be a definite aid as regards strength and watertightness. Since the layer is thin, optical means are necessary for its identification and considerable magnification is needed to see it. The congealed layer that Mr. Anderegg speaks of is a layer of some magnitude.

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[2] President, Hy-Test Cement Co., Philadelphia, Pa.

MR. MALMED.—Call it what you please but Mr. Anderegg's theory is entirely opposed to Mr. Voss's theory. Mr. Voss construes the bond layer as a benefit and Mr. Anderegg construes it as an absolute detriment.

MR. F. O. ANDEREGG.[3]—Mr. Malmed has misinterpreted part of the conclusion. My findings are: A highly absorbent, unwetted brick congeals the surface of mortar before it has a chance to flow out over and come in intimate contact with the surface of the brick. If lime is present in the mortar, however, part of the lime is carried to the interface by the moisture as it is drawn into the brick. The amount carried depends upon the lime content of the mortar. The presence of this lime at the interface is helpful in reducing leakage and the separation of wet joints by freezing. A certain amount of lime, in combination with a minimum suction rate, seems to be required to produce what I have spoken of as a "congealed layer." This may be the same as Mr. Voss's "bond layer." My experience with masonry cements has rarely found one which would form such a congealed layer, although such mortars do lose water and plasticity in the layer next the interface very quickly. To observe whether such a "bond layer" or "congealed layer" is being formed, throw some of the given mortar on the flat side of a very absorbent brick and remove the soft part of the mortar within a few seconds.

MR. C. C. CONNOR.[4]—Mr. Anderegg gives optimum figures of 20 g. for the rate of absorption for brick with a 1:1:6 mortar and 30 g. with a 1:2:9 mortar. In field practice I have had to establish 25 g. as an upper limit for 1:1:6 and 1:2:7½ mortars which would indicate much lower optimums. A 5 to 25-g. range, when used with high-lime mortars, has produced more than 40 consecutive buildings which have been watertight for a period of from 1 to 11 yr. and seems to be well established as satisfactory. It would be very helpful if higher rates of absorption could be used with consistent success, but the indications in the field are that such brick increase the separation cracking and are liable to cause leaks. The high rate of absorption bricks must be wetted to give any hopes of good results, and wetting is an uncertain practice.

In his verbal discussion, Mr. Anderegg explained how he obtained consistent results in his experiments by coating the bricks on each side of the joint with mortar and then putting the two mortar coatings together to form the joint. Such a practice is vastly different from that used in the field and might very well reduce the detrimental effect of a high rate of absorption. Mr. Hardesty, of the Bell Telephone Laboratories, found in some experimental work (unpublished) that he could obtain a considerable betterment in the extent of bond to brick if he wiped the surfaces of the brick with a thin coat of the mortar material before laying the brick. I should like to ask Mr. Anderegg whether it is not possible that his method of applying the mortar to the bricks might not have produced these high optimum rates of absorption.

Palmer and Parsons[5] found that the best results in bond durability were obtained by keeping the rate of absorption of bricks below 40 g. But they arrived at that conclusion from data obtained by laying the brick on a mortar bed for one brick only and throwing the operator's weight upon each brick as it was laid. It would seem that the practices of Messrs. Palmer and Parsons and Mr. Anderegg achieved a quite similar result.

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[3] Consulting Specialist on Building Materials, Newark, Ohio.

[4] Assistant Engineer, New Jersey Bell Telephone Co., Newark, N. J.

[5] L. A. Palmer and D. A. Parsons, "A Study of the Properties of Mortars and Bricks and Their Relation to Bond," *Journal of Research*, Nat. Bureau Standards, Vol. 12, No. 5, May, 1934, p. 609 (*RP. No. 683*).

Mr. Anderegg's work is a very pertinent corroboration of Palmer and Parsons' conclusions that the rate of absorption of brick has a great effect upon bond, and that there are definite limits to the rate of absorption outside of which good results cannot be expected. This has been a basic premise in the masonry specifications used in my company for many years and has been amply borne out by field results. The poor results that Mr. Anderegg obtained with very low absorption brick also bear out field results where a high amount of separation cracking occurs with such brick. Very low suction apparently has been a contributing cause of the condition commonly known as hair cracking which causes leaks and is extremely expensive to remedy by ordinary moistureproofing practices.

The fact that the contact surface shown in the photograph was closely molded to the very low absorption brick without obtaining a good bond was rather unexpected. Bonds that are apparently good and also durable have been obtained in the field between high-lime mortars and the glazed ends of bricks, although the horizontal surfaces of the bricks must have a proper suction. The low strength of the bonds obtained by Mr. Anderegg with good contact is a factor that must be considered if a test for extent of bond is ever developed.

There is no question of the importance of the evidence given in this paper concerning the difference in results produced by variations in the rates of absorption of brick and the kind of mortar mixes used. Transposition of laboratory data into field specifications, however, must be done with caution in the masonry field where the manner of laying the masonry units has such a great effect upon bond. I believe that the optimum rates of absorption of brick to produce water-tight brick bonds under ordinary field conditions and practices are considerably lower than those given by Mr. Anderegg. However, I would expect that the different mortar mixes would maintain approximately the same relative differences as shown in this paper.

MR. ANDEREGG.—Mr. Connor is interested in the optimum absorption rate for different mortars. The following values have been reported to the American Ceramic Society[6]: 10 g. for a 2:1:9 mortar; 20 g. for a 1:1:6 mortar, which is the one I understand Mr. Connor likes to use; and 30 g. 1:2:9 mortar, which is highest of these in lime. In that same paper scatter results were reported for the absorption rates of bricks taken from a given kiln course, or altitude zone. One sample contained bricks which absorbed from 30 to 90 g. during the first minute. About half of these took up between 45 and 70 g. Such wide variations in absorption rates indicate that there may be some practical difficulty in applying close specifications. However, Mr. Connor's proposal will help to bring to the attention of the brick manufacturers a fine opportunity to contribute to the integrity of brick walls, by producing bricks of better absorption characteristics.

MR. J. C. PEARSON.[7]—In recent months we have been giving some study in our laboratory to different methods of test for measuring the bond between mortar and bricks, and in most cases we have found very wide variations in the results from similar specimens. Will you please describe the method you use, and indicate what order of reproducibility is attained?

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[6] Paper not yet published; is being prepared for publication.

[7] Director of Research, Lehigh Portland Cement Co., Allentown, Pa.

MR. ANDEREGG.—The method was described two years ago. It consists of using a very meticulous technique to bring the mortar into complete contact with the brick. The mortar is placed on each brick and wiped around all four sides, and the excess is scraped off. Then these bricks are put together and pushed down to $\frac{1}{2}$ in. They are set aside and watched carefully, and at that particular point, which approximates roughly the initial set—anyone that has worked with it will know it—they are carefully tooled with a round tool, bringing the mortar into intimate contact with the corners of both bricks. That has to be done very carefully, putting your finger or thumb over the mortar at the corner and then working away from it with the tool, so as not to break off the corner.

On the second day, these joints are painted with a water-soluble dye. When they are broken, if there is more than a very slight trace of dye penetration, those results are discarded. We did not make any attempt to correct for them. The results reported here were obtained with the help of an excellent assistant and it was extremely rare that we had any bonds that did not make complete contact.

MR. PEARSON.—That brings up the fundamental question of whether the indications from the meticulous laboratory test will be relatively the same as from the bricklayer's manipulation on the job. I do not know the answer, but I imagine the laboratory test would tend to give relatively higher bond for the weaker and less plastic mortars. If this is true, then it would seem preferable to have some sort of test for the bond of a clean brick laid on a bed of mortar. This is what we have been trying to do, and I think we have made some progress, but it is difficult to develop the degree of mechanical control that will insure consistent and reproducible results.

MR. ANDEREGG.—Mr. Pearson reports poor success in getting reproducible results. That was also our experience at first until we used the dye to find the causes. It took us several months to work out a technique and train ourselves to bring the scatter within 5 per cent. When the contact is incomplete, a reentrant angle or discontinuity occurs, at the apex of which is concentrated a high stress. The magnitude of the stress depends upon the acuteness of the angle of contact and upon the distance of the apex from the surface of the brick. In other words, a notched specimen is under test with unknown notch dimensions.

Almost all masons whom I have contacted have had to receive special coaching before they would manipulate the mortar to secure complete contact. This experience is in accord with the feeling of Connor and Pearson in questioning the extrapolation of these results to practical construction. It is of interest to note, however, that the bond data given here have been running about four times as high as those reported in 1931.[8] The earlier results were obtained by breaking panels or columns in flexure. They were built by several brick masons and the mortars also ranged from straight lime to straight portland cement. The scatter among the older data was, of course, very much higher.

MR. NORMAN W. KELCH.[9]—I would like to ask Mr. Anderegg whether he noticed any difference between the bond to the upper brick and the lower brick as laid. Subsequent to the 1928 series of tests we conducted at the University of California, we built piers about 28 in. high, some of which we loaded by means of steel plates on the top and bottom connected with long bolts. The nuts were tightened each day in a calculated amount to simulate a scaffold high of brickwork being superimposed each day until the equivalent of a four-story building was reached. The loaded piers gave modulus of rupture values higher than those not loaded and we noticed that the mortar invariably stuck to the upper brick as laid.

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[8] F. O. Anderegg, "Watertight Brick Masonry," *Architectural Record*, p. 208 (1931).

[9] Engineer-Manager, Clay Products Association of the Southwest, Austin, Tex.

I was wondering if you had noticed that in your specimens.

MR. ANDEREGG.—We have broken several hundred two-brick beams and have noted about as many breaks next the bottom brick as against the top brick.

MR. STALEY.—The failure at the bottom of the mortar joint can be accounted for by loss of water. Since the mortar is placed on the bottom brick, there is some opportunity for loss of water from the mortar to the brick before the upper brick is placed and this loss of water takes place while there is no pressure on the mortar joint. The mortar is in a more plastic state at the upper side than at the lower when the top brick and the pressure are applied, and thus a more intimate and stronger contact is obtained at the upper brick surface.

MR. ANDEREGG.—Since the presentation of my paper at the annual meeting, one brick of each of types *B*, *E*, and *S* was sent to Mr. R. V. Stull of the National Bureau of Standards for the determination of the permeabilities and other pore properties by his methods.[10] Grateful acknowledgement is due to Mr. Stull for this courteous cooperation. The results reported by him are given in the accompanying Table I.

TABLE I.—PORE PROPERTIES OF SINGLE SPECIMENS OF BRICK TYPES *B*, *E*, AND *S*.

Brick Type	Air Permeability, $f \times 10^6$	Capillary Radius, r, μ	Number of Capillaries, $N \times 10^{-3}$	Absorption, per cent[a]				Saturation Coefficients		Pores Filled by 5-hr. Boiling, per cent[c]
				by Weight		by Volume				
				(1)	(2)	(3)	(4)	(1)/(2)	(3)/(4)	
B..........	2 213	1.10	6 674	10.95	13.23	21.80	26.91	0.83	0.81	97.6
E..........	679	0.59	25 900	10.62	13.41	20.38	28.47	0.79	0.72	91.1
S..........	159 400	8.46	140	12.78	19.29	22.37	32.04	0.66[b]	0.70	106.1

[a] (1) = Per cent by weight of water absorbed by 24-hr. submersion.
(2) = (1) followed by 5-hr. boiling and cooling.
(3) = (1) expressed in volume per cent.
(4) = Per cent porosity of specimen.

[b] Brick *S* apparently took up more water than was required to fill the pores, indicating hydration.

[c] These per cent values $\doteq 100\ ((3)/(4))/((1)/(2))$.

Brick *S* has a permeability about 220 times as great as that of brick *E* and yet a lime mortar set up or stiffens faster on *E* than on *S*. This may be ascribed to two phenomena,—the formation of a dense, congealed layer of lime at the interface of brick *S*, and the smaller suction force exerted by the larger capillaries of brick *S*. Water is drawn up about 14 times as high in a tube having a diameter of 0.6 micron as in one with a diameter of 8.5 microns.

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[10] Ray T. Stull and Paul V. Johnson, "Some Properties of the Pore System in Bricks and their Relation to Frost Action," *Journal of Research*, Nat. Bureau Standards, Vol. 25, p. 711 (1940).

RELATIONS BETWEEN RESULTS OF LABORATORY FREEZING AND THAWING AND SEVERAL PHYSICAL PROPERTIES OF CERTAIN SOFT-MUD BRICKS*

By John W. McBurney[1]

Synopsis

A sample of 50 bricks representative of the production of 10 plants making soft-mud bricks from surface clay was subjected to the various tests prescribed by the current A.S.T.M. specifications. Additional tests were made using the methods of Stull and Johnson. Freezing-and-thawing tests were made until 175 cycles were completed. The results of 50 cycles and 175 cycles were compared and the relations between these results and the previously measured properties were considered. The relation between results of freezing and thawing and properties is much more definite for 175 cycles than for 50 cycles.

Analyses of the data presented in BMS 60[2] show that relatively few of the soft-mud bricks from the New England district failed in 50 cycles of freezing and thawing according to A.S.T.M. Standard Methods for Sampling and Testing Brick (C 67 – 41),[3] even though some of these bricks had high absorptions, high saturation coefficients, and relatively low strengths, and were classified by the manufacturers as "light hard," "interior," or "soft." The failures that did occur during 50 cycles were, in nearly all instances, on bricks failing to meet the physical requirements for grade *SW* of A.S.T.M. Tentative Specifications for Building Brick (Made from Clay or Shale) (C 62 – 41 T).[4] Other samples of these high absorption, soft-mud bricks performed poorly in exposure tests. These discrepancies between the results of 50 cycles of freezing and thawing, on the one hand, and the manufacturer's classifications, the physical properties usually effective in separating durable from nondurable bricks and the results of exposure tests, on the other hand, warrant consideration of the appropriateness of the specification requirements[4] for freezing and thawing. This is particularly true because passing 50 cycles of freezing and thawing is accepted as a waiver or replacement of the physical requirements of grade *SW*. A secondary purpose of the present paper is to consider the utility of certain nonspecification properties of brick as measures of durability.

Samples

Through the cooperation of Frederick Heath, Jr.,[5] five bricks from each of ten

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* The presentation of this paper at the annual meeting was sponsored by Committee C-15 on Manufactured Masonry Units.

[1] Senior Technologist, National Bureau of Standards, Washington, D. C.

[2] J. W. McBurney and J. C. Richmond, "Strength, Absorption and Resistance to Laboratory Freezing and Thawing of Building Bricks Produced in the United States," *Building Materials and Structures Report BMS 60*, Nat. Bureau Standards, November 5, 1940.

[3] 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 18.

[4] *Ibid.*, p. 267.

[5] Secretary-Engineer, Colonial Clays, Inc., Worcester, Mass.

TABLE I.—WATER ABSORPTIONS BY 24-HR. COLD IMMERSION AND BY 5-HR. BOILING, SATURATION COEFFICIENTS, TRANSVERSE AND COMPRESSIVE STRENGTHS, AND RESULTS OF LABORATORY FREEZING AND THAWING AT 50 AND 175 CYCLES ON 50 SOFT-MUD, SURFACE-CLAY BUILDING BRICK.

Brick		Water Absorption, per cent				Saturation Coefficient (C/B)		Modulus of Rupture, psi.	Compressive Strength, psi.	Results of Freezing and Thawing, Weight Loss, per cent, or Number of Cycles to Failure, F-c	
		24-hr. Cold Immersion		5-hr. Boiling							
Plant	Course in Kiln	Test M[a]	Test N[b]	Test M[a]	Test N[b]	Test M[a]	Test N[b]			50 Cycles Method B	125 Additional Cycles Method 3
1	*2*	*3*	*4*	*5*	*6*	*7*	*8*	*9*	*10*	*11*	*12*
A	17	4.7	4.9	7.3	7.9	0.64	0.62	1 210	7 650	0.21	0.30
	25	6.1	6.1	7.7	8.1	0.80	0.76	1 500	12 600	0.19	0.41
	29	3.3	3.3	4.6	6.7	0.72	0.50	1 520	11 400	0.10	0.20
	33	4.3	4.5	6.5	8.1	0.66	0.56	1 480	9 750	0.11	0.15
	45	16.7	16.9	18.3	18.0	0.91	0.94	1 070	4 950	0.20	F70c
B	18	6.3	6.4	9.9	10.0	0.64	0.64	720	9 700	0.21	0.38
	22	9.1	9.3	13.6	13.2	0.67	0.70	865	5 950	0.14	0.23
	30	13.6	13.8	18.5	17.9	0.73	0.77	635	4 050	0.12	F121c
	42	17.8	18.2	22.3	21.3	0.80	0.85	560	2 650	0.41	F63c
	46	19.8	20.2	24.0	22.7	0.83	0.89	425	2 000	0.50	F85c
C	17	8.3	9.0	11.9	12.1	0.69	0.74	1 575	7 050	0.12	0.34
	34	13.3	13.4	16.5	16.5	0.80	0.81	1 210	5 400	F45c	....
	41	15.4	15.0	18.6	18.7	0.83	0.81	915	4 750	0.25	F71c
	45	16.4	16.5	18.9	18.9	0.86	0.87	550	3 750	0.25	F65c
	49	18.4	18.3	20.7	20.2	0.89	0.91	320	2 800	1.33	F73c
D	6	13.0	13.2	16.3	15.7	0.80	0.84	1 070	5 850	0.22	F91c
	18	9.6	9.8	12.7	12.2	0.76	0.80	1 105	5 500	0.24	0.41
	22	11.7	12.0	15.4	15.0	0.76	0.80	1 305	6 000	0.14	F144c
	37	16.1	16.1	20.2	19.6	0.80	0.83	940	4 250	0.26	F62c
	45	17.8	17.7	21.9	21.2	0.81	0.84	545	2 800	0.38	F94c
E	13	12.1	12.3	15.3	15.1	0.79	0.82	860	5 400	F24c	....
	22	9.0	9.1	12.4	12.0	0.73	0.76	1 320	9 200	0.23	0.26
	33	14.4	14.4	17.7	17.5	0.81	0.83	1 060	5 100	0.70	F63c
	42	17.4	17.9	20.6	20.4	0.85	0.88	980	4 300	1.12	F65c
	49	19.2	19.4	20.1	20.0	0.96	0.97	550	2 200	0.65	F108c
F	17	13.7	13.9	18.7	18.4	0.73	0.76	275[c]	5 050	0.31	F131c
	29	18.7	17.3	22.7	21.0	0.82	0.83	485[c]	5 000	F46c	....
	33	14.5	13.3	19.7	17.8	0.74	0.75	1 380	4 700	2.55	F122c
	41	18.4	17.4	22.3	20.2	0.82	0.86	955	4 150	2.25	F71c
	46	18.8	17.9	22.4	20.0	0.84	0.90	755	3 750	0.60	F117c
G	17	9.7	9.8	12.2	12.0	0.79	0.81	1 600	6 400	F49c	....
	26	5.8	6.3	6.3?	8.1	0.93?	0.78	1 540	9 350	0.11	0.36
	33	15.3	15.5	18.7	18.5	0.82	0.84	860	5 900	F17c	....
	41	14.8	15.4	18.7	18.5	0.79	0.83	980	5 500	F24c	....
	49	15.8	16.4	19.2	18.6	0.82	0.89	1 300	4 550	F45c	....
H	6	12.8	13.1	16.0	16.0	0.80	0.82	1 470	4 900	F37c	....
	13	12.0	12.2	15.0	15.0	0.80	0.81	940	4 750	....	F91c
	21	14.0	14.5	17.5	17.5	0.80	0.83	1 320	4 800	F27c	....
	34	14.1	14.4	16.6	16.5	0.84	0.87	1 320	4 550	0.25	F109c
	45	12.3	12.8	16.2	16.1	0.76	0.80	1 400	4 700	0.13	F113c
I	14	3.4	3.6	5.6	6.3	0.61	0.57	1 470	8 550	0.13	0.21
	26	10.9	11.2	13.8	13.0	0.79	0.86	1 240	7 900	0.46	0.56
	29	15.4	15.6	18.7	18.1	0.82	0.86	840	4 400	1.34	F83c
	42	17.2	16.5	19.6	18.0	0.88	0.92	780	3 950	0.49	F128c
	50	17.3	17.8	19.2	18.5	0.90	0.96	830	3 450	0.37	F87c
J	14	15.1	15.9	19.3	18.8	0.78	0.85	980	5 350	0.40	F69c
	26	14.7	14.6	18.5	18.0	0.80	0.81	1 020	5 450	F47c	....
	38	17.6	17.3	21.1	20.8	0.83	0.83	845	4 250	0.36	F82c
	45	17.6	17.5	21.2	20.9	0.83	0.84	810	4 050	0.37	F60c
	49	19.4	19.3	22.0	21.9	0.88	0.88	590	2 250	0.37	F59c
Avg.		13.46	13.54	16.66	16.35	0.795	0.809	....	....	....	....

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[a] Tests on whole bricks made at National Bureau of Standards.

[b] Tests on whole bricks made in laboratory of Colonial Clays, Inc.

[c] Cracks in brick.

TABLE II.—WATER ABSORPTION BY 1-MIN. COLD IMMERSION, POROSITY BY AIR EXPANSION METHOD' SATURATION COEFFICIENT, (*W*/*V*), CALCULATED FROM POROSITY, AIR PERMEABILITY, NUMBER OF PORES PER SQUARE CENTIMETER AND AVERAGE RADIUS OF PORES CALCULATED FROM POROSITY AND PERMEABILITY AND RESULTS OF LABORATORY FREEZING AND THAWING AT 50 AND 175 CYCLES ON 50 SOFT-MUD, SURFACE-CLAY BUILDING BRICK.

Brick		Water Absorption, 1-min. Cold Immersion, per cent	Porosity, per cent	Saturation Coefficient from Porosity[a] (*W*/*V*)	Ratio, Pores Filled by Boiling to Porosity[b] (*V*), (*W*/*V*)/(*C*/*B*)	Permeability to Air,[c] $f \times 10^4$	Number of Capillaries per Square Centimeter,[d] $N \times 10^{-4}$	Average Radius of Capillaries,[e] r, μ	Results of Freezing and Thawing, Weight Loss, per cent, or Number of Cycles to Failure, F-c	
Plant	Course in Kiln								50 Cycles Method B	125 Additional Cycles Method 3
1	*2*	*3*	*4*	*5*	*6*	*7*	*8*	*9*	*10*	*11*
A	17	0.7	19.4	0.52	0.81	5.00	1 634	0.615	0.21	0.30
	25	1.2	21.0	0.62	0.78	4.06	2 357	0.533	0.19	0.41
	29	0.6	15.7	0.47	0.65	4.05	1 322	0.608	0.10	0.20
	33	0.7	18.7	0.50	0.75	3.99	1 894	0.560	0.11	0.15
	45	5.9	36.6	0.80	0.87	4.91	5 920	0.444	0.20	F70c
B	18	2.0	23.2	0.56	0.88	19.76	591	1.12	0.21	0.38
	22	3.4	27.7	0.63	0.94	33.13	503	1.33	0.14	0.23
	30	6.8	33.6	0.72	0.98	39.86	616	1.32	0.12	F121c
	42	9.7	38.9	0.76	0.95	22.84	1 438	0.929	0.41	F63c
	46	8.9	41.6	0.76	0.92	13.43	2 798	0.689	0.50	F85c
C	17	3.2	28.6	0.56	0.80	4.80	3 670	0.496	0.12	0.34
	34	6.5	34.6	0.68	0.84	7.07	3 675	0.548	F45c	....
	41	7.5	37.2	0.71	0.86	7.08	4 241	0.529	0.25	F71c
	45	7.8	37.4	0.74	0.86	6.71	4 522	0.514	0.25	F65c
	49	8.0	36.8	0.82	0.93	6.84	4 305	0.522	1.33	F73c
D	6	6.0	33.4	0.69	0.87	12.52	1 931	0.742	0.22	F91c
	18	2.8	29.1	0.62	0.82	8.77	2 096	0.665	0.24	0.41
	22	4.0	33.0	0.64	0.84	10.90	2 169	0.697	0.14	F144c
	37	8.8	37.0	0.74	0.93	22.01	1 349	0.743	0.26	F62c
	45	9.6	39.0	0.77	0.94	20.23	1 631	0.694	0.38	F94c
E	13	4.2	32.8	0.67	0.84	10.18	2 292	0.675	F24c	....
	22	2.1	28.5	0.60	0.83	8.37	2 106	0.656	0.23	0.26
	33	6.5	35.9	0.70	0.86	8.22	3 403	0.580	0.70	F63c
	42	8.0	40.2	0.72	0.84	5.22	6 700	0.437	1.12	F65c
	49	8.5	41.4	0.76	0.79	2.52	14 760	0.299	0.65	F108c
F	17	6.9	34.8	0.68	0.93	....[f]	....	....	0.31	F131c
	29	12.7	39.4	0.77	0.94	....[f]	....	....	F46c	....
	33	7.4	36.4	0.68	0.92	1.75	16 420	0.266	2.55	F122c
	41	10.7	41.1	0.73	0.88	1.11	33 010	0.199	2.25	F71c
	46	10.8	40.9	0.75	0.89	1.23	29 510	0.210	0.60	F117c
G	17	1.9	29.8	0.61	0.77	0.176	109 500	0.093	F49c	....
	26	0.6	22.2	0.54	0.58	0.186	57 500	0.099	0.11	0.36
	33	6.8	37.5	0.70	0.85	0.285	107 100	0.106	F17c	....
	41	7.0	37.9	0.66	0.84	0.251	124 200	0.099	F24c	....
	49	6.3	38.8	0.68	0.83	0.128	255 100	0.070	F45c	....
H	6	4.8	35.1	0.65	0.82	0.147	181 900	0.078	F37c	....
	13	4.3	33.3	0.66	0.83	0.171	140 700	0.087	0.00	F91c
	21	5.7	37.2	0.66	0.82	0.166	181 000	0.081	F27c	....
	34	5.7	35.0	0.71	0.84	0.212	125 400	0.094	0.25	F109c
	45	4.7	35.6	0.62	0.81	0.160	172 000	0.081	0.13	F113c
I	14	1.2	19.0	0.37	0.61	0.198	39 590	0.124	0.13	0.21
	26	4.2	30.9	0.65	0.82	0.500	41 450	0.154	0.46	0.56
	29	7.3	36.8	0.72	0.87	0.791	37 150	0.178	1.34	F83c
	42	8.2	39.9	0.72	0.82	0.330	104 700	0.110	0.49	F128c
	50	7.6	40.9	0.70	0.78	0.264	137 500	0.097	0.37	F87c
J	14	7.9	38.3	0.66	0.85	0.493	64 600	0.138	0.40	F69c
	26	7.6	36.7	0.68	0.86	0.608	48 080	0.156	F47c	....
	38	9.8	39.8	0.72	0.87	0.610	56 350	0.150	0.36	F82c
	45	9.6	40.6	0.70	0.85	0.583	61 350	0.145	0.37	F60c
	49	10.6	41.7	0.74	0.84	0.456	82 750	0.127	0.37	F59c

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[a] Ratio of percentage of porosity (*W*) calculated from water absorption by 24-hr. cold immersion to percentage of porosity (*V*) measured by the volumetric method.

[b] Ratio of saturation coefficient (*W*/*V*) to saturation coefficient (*C*/*B*). Percentage pore space filled by boiling = 100 (*W*/*V*)/(*C*/*B*).

[c] The permeability f equals QT/AP where Q = number of cubic centimeters of air passing per second through the brick; P = pressure difference across the brick in grams per square centimeter; A = average cross-sectional area in square centimeters over which flow takes place; and T = thickness of the brick, in centimeters, through which transmission of the air occurs.

[d] The number of capillaries N equals $21.7\ V^2/f$ where V is the percentage of porosity, and f is the air permeability as defined in note [c].

[e] The mean effective radius r in microns equals $0.012\ (f/V)^{1/2}$, where f is the air permeability and V is the percentage of porosity.

[f] Cracked bricks.

New England brick plants (A to J) were provided. All ten of these plants manufacture bricks from surface clay by the soft-mud process and fire in updraft (scove) kilns. Each sample represented a range from bottom to top of the kiln and the individual bricks are numbered according to the course from which they were taken.

Test Methods and Presentation of Data

Table I presents water absorption by 24-hr. cold immersion and by 5-hr. boiling, saturation coefficient (C/B) calculated from these absorptions, and also modulus of rupture and compressive strength for all bricks. These tests were made in accordance with Methods C 67 – 41.[3] The columns headed N report the results of tests made at Mr. Heath's laboratory on whole-brick specimens, whereas the M results were obtained on the same whole bricks at the National Bureau of Standards. The left-hand column under the heading, "Results of Freezing and Thawing," records either the loss in weight as a percentage of the dry weight or the number of cycles causing failure (F-c) up to 50 cycles of method B of Methods C 67 – 41.[3] The second column under the same heading reports the results of an additional 125 cycles made according to method 3 of the author's 1938 paper.[6] This method differs from method B in that the specimens remain totally immersed in water for the 48 hr. between groups of 5 cycles whereas with method B the period is used for air drying.

Table II presents further data on these same bricks. These data include water absorption by 1-min. total immersion, porosity measured by the gas expansion method, a saturation coefficient (W/V) consisting of the ratio of porosity measured by 24-hr. immersion in cold water to porosity by the gas expansion method, the ratio of W/V to C/B, the calculated number of capillaries per square centimeter of the brick, and the calculated average radius of capillaries. All data except those presented in columns 3, 10, and 11 were obtained using the apparatus and methods described by Stull and Johnson.[7] The data from Table I for freezing and thawing results are reproduced in Table II for convenience in comparing relations.

Discussion of Data

Reproducibility of Tests:

The M and N columns in Table I provide comparisons of tests made by one laboratory with those made by a different laboratory on the same bricks. The agreement is not so good as that reported by Krefeld[8] for a similar comparison. An explanation offered for this relatively poor agreement is that these New England brick have very fine pore structures as shown by comparing the average radius of capillaries reported in Table II with the corresponding measurements reported by Stull and Johnson.[7] Since the 5-hr. boiling test produces only a partial filling of the pores of such brick, low ratios obtain for the porosity measured by 5-hr. boiling to porosity measured by the gas-expansion method. Furthermore, the degree of filling the pores of such bricks by absorption methods varies with small differences in the technique used. The poor reproducibility of absorption tests on bricks with $(W/V)/(C/B)$ ratios less than unity has been previously noted.[9]

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[6] J. W. McBurney and A. R. Eberle, "Freezing and Thawing Tests for Building Brick," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part II, p. 470 (1938).

[7] R. T. Stull and P. V. Johnson, "Some Properties of the Pore System in Bricks and Their Relation to Frost Action," *Journal of Research*, Nat. Bureau Standards, Vol. 25, December, 1940, p. 711 (*RP 1349*).

[8] W. J. Krefeld and J. W. McBurney, "Comparison of Standard Tests on Building Brick by Two Laboratories," ASTM Bulletin, No. 96, January, 1939, p. 7.

The averages for these absorptions and saturation coefficients (C/B) are given in Table I. The mean deviations are 0.34 per cent for water absorption by 24-hr. cold immersion, 0.62 per cent for absorption by 5-hr. boiling and 0.038 for the saturation coefficient. Although there appear to be systematic differences in the results since the saturation coefficients determined by laboratory *N* are greater in 39 tests and less in only 6 tests than are the corresponding saturation coefficients determined by laboratory *M*, there is no evidence that one series of tests is more accurate than the other. The figure for water absorption by 5-hr. boiling and the corresponding saturation coefficient reported by laboratory *M* for brick specimen G26 is probably in error since other results, such as porosity and resistance to freezing and thawing indicate a lower absorption. Unless otherwise noted, subsequent discussions will be limited to data resulting from tests by laboratory *M*.

TABLE III.—COMPARISON OF RESULTS OF 50 AND 175 CYCLES OF FREEZING AND THAWING WITH ABSORPTIONS, SATURATION COEFFICIENTS AND STRENGTHS OF 50 SOFT-MUD, SURFACE-CLAY BUILDING BRICKS.

P = passed, F = failed, T = total bricks in range.

Property and Range in Property	Number of Cycles	Range Within Which All Bricks Passed[a]	Number of Bricks in Range, P	Range Within Which Bricks Both Passed and Failed[a]	Number of Bricks			Range Within Which All Bricks Failed[a]	Number of Bricks, F
					P	F	T		
Water Absorptions:									
1-min. cold immersion (0.6 to 12.7 per cent)	50	0.6 to 1.2	6	1.9 to 10.8	29	14	43	12.7[b]	1
	175	0.6 to 1.2	6	1.9 to 4.2	6	3	9	4.3 to 12.7	35
24-hr. cold immersion (3.4 to 19.8 per cent)	50	3.4 to 9.6	11	9.7 to 19.8	24	15	39	[c]	..
	175	3.4 to 9.6	11	9.7 to 10.9 (*M*) 9.8 to 11.2 (*N*)	1	1	2	11.7 to 19.8	37
5-hr. boiling (4.6 to 24.0 per cent)	50	4.6 to 11.9	8	12.2 to 24.0	27	15	42	[d]	..
	175	4.6 to 11.9	8	12.2 to 13.8 (*M*) 12.0 to 13.2 (*N*)	4	1	5	15.0 to 24.0	37
Saturation Coefficients:									
C/B (0.61 to 0.96)	50	0.61 to 0.73	10	0.74 to 0.96	25	15	40	[e]	..
	175	0.61 to 0.72	7	0.73 to 0.80(*M*) 0.75 to 0.80 (*N*)	5	17	22	0.81 to 0.96	21
W/V (0.37 to 0.82)	50	0.37 to 0.60	8	0.61 to 0.80	27	14	41	0.82[f]	1
	175	0.37 to 0.60	8	0.61 to 0.65	4	4	8	0.66 to 0.82	34
Strengths:									
Modulus of rupture (275 to 1600 psi.)	50	[g]	..	275 to 1600	35	15	50	[g]	..
	175	[g]	..	275 to 1600	12	38	50	[g]	..
Compressive strength (2000 to 12 600 psi.)	50	7050 to 12 600	10	2000 to 6400	25	15	40	[h]	..
	175	7050 to 12 600	10	5500 to 6400	2	5	7	2000 to 5450	33
Porosity:									
(volume) (15.7 to 41.6 per cent)	50	5.7 to 29.1	11	29.8 to 41.7	24	15	39	[i]	..
	175	15.7 to 29.1	11	29.8 to 30.9	1	1	2	32.8 to 4.7	37

[a] The criterion for passing is no breakage or loss in weight not exceeding 1.0 per cent at stated number of cycles.
[b] The brick with the next highest absorption (10.8 per cent) passed.
[c] The brick with the highest absorption (19.8 per cent) passed.
[d] The brick with the highest absorption (24.0 per cent) passed.
[e] The brick with the highest saturation coefficient C/B (0.96) passed.
[f] The brick with the highest saturation coefficient W/V (0.82) passed.
[g] Failures occurred at random throughout the entire range including the brick with highest modulus of rupture (1600 psi.).
[h] The brick with the lowest strength passed.
[i] The brick with the highest porosity passed.

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[9] Joseph C. Richmond and John W. McBurney, "Effect of Freezing Temperature in Freezing-and-Thawing Tests of Brick," *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 967 (1941).

Relations Between Water Absorptions and Resistance to Freezing and Thawing:

From inspection of the data presented in Tables I and II, it is evident that the results of freezing and thawing are related to the water absorptions of the bricks. Considering the results of 175 cycles, there is a range of low absorptions where no failures take place, an intermediate range where both failures and passes occur, and a high range where all brick fail. For the purposes of this paper, a brick was considered as having failed when it became broken or underwent a 1 per cent loss of its dry weight. Table III presents these ranges fixed by the results of 50 and 175 cycles and indicates the number of specimens occurring in each range

The results of both 50 and 175 cycles fix definite absorption limits (1.2 per cent for 1-min. immersion, 9.6 per cent for 24-hr. immersion, and 11.9 per cent for 5-hr. boiling) under which no failures occur. Above these limits the results of 50 cycles are practically random mixtures of passing and failing. The effect of 175 cycles is to lower the upper limit of this indeterminate range and to establish a new range within which all bricks fail. In other words, 50 cycles selects the absorption limit below which no failures take place as effectively as is done by 175 cycles, but above this limit a more severe test is required to differentiate between more durable and less durable bricks.

Relation Between Saturation Coefficients and Resistance to Freezing and Thawing:

Saturation coefficients calculated either from absorption by 5-hr. boiling (C/B) or from porosity determined by the gas-expansion method (W/V) closely resemble absorptions in the manner in which they distinguish durable from nondurable brick as measured by results of freezing and thawing. The effect of 175 cycles compared with 50 cycles was to lower the saturation coefficient (C/B) associated with a failure from 0.74 to 0.73. At 50 cycles the range 0.74 to 0.96 was random and indeterminate although only one failure occurred below 0.78. For 175 cycles, all bricks with C/B ratios (saturation coefficients) exceeding 0.80 failed, 12 out of 15 failed in the range 0.78 to 0.80, and 2 passed and 5 failed between 0.73 and 0.77. The two bricks (B30 and F17) which failed with saturation coefficients of 0.73 measured by laboratory M had coefficients of 0.77 and 0.76, respectively, according to laboratory N.

All of the bricks included in this investigation are of such nature that 5-hr. boiling does not completely fill the pores and these bricks therefore have saturation coefficients (W/V) which are significantly lower than those measured by boiling (C/B). The total range for W/V ratios is 0.37 to 0.82 whereas the corresponding C/B ratios range from 0.61 to 0.96. Considering the results of 175 cycles of freezing and thawing, all bricks with W/V values of 0.60 or less passed and all bricks in the range from 0.66 to 0.82 (maximum) failed. The 8 bricks in the indeterminate range 0.61 to 0.65 classified as 4 passes and 4 failures. With the exception of B22 (W/V of 0.63, C/B of 0.67 and $(W/V)/(C/B)$ of 0.94) all of these 8 bricks had C/B ratios in the range 0.76 to 0.80. The apparently greater precision of the W/V ratio compared with the C/B ratio in separating durable from nondurable bricks measured by the results of 175 cycles may, in part, be explained by the greater accuracy and reproducibility of measurements by the gas-expansion method on this type of brick. The available data provide no information as to whether the zone dividing durable from nondurable bricks would conform to the numerical limits for the C/B ratio or for the W/V ratio if freezing-and-thawing tests, corresponding in severity to the 175 cycles here reported, were used on brick samples with $(W/V)/(C/B)$ ratios of unity.

Relation Between Strengths and Resistance to Freezing and Thawing:

Modulus of rupture is not related to durability according to the data here presented. The brick (G17) with the highest modulus of rupture (1600 psi.) failed in less than 50 cycles. Brick F17 required 70 cycles to produce failure and had a modulus of rupture of 275 psi. Passes and failures occurred at random between these limits. Compressive strength, however, compares favorably with absorption and the saturation coefficients in its relation to the results of 175 cycles of freezing and thawing. All bricks with compressive strengths of less than 5500 psi. failed. All bricks exceeding 6400 psi. passed. The intermediate range included 7 bricks of which 2 passed and 5 failed.

Relations of Porosity, Air Permeability and Pore System to Resistance to Freezing and Thawing:

The values of porosity, determined by the gas-expansion method, are excellently related to the results of 175 cycles of freezing and thawing. The precision with which this porosity measurement separates durable from nondurable brick is equalled only by the results of water absorption by 24-hr. cold immersion. The overlapping range for porosity (29.8 to 30.9 per cent) includes the same two bricks (G17 and I26) which are included in the corresponding overlapping range for absorption by 24-hr. cold immersion (9.7 to 10.9 per cent).

The values for air permeability, expressed as $f \times 10^4$, range from 0.128 to 39.86 and are not correlated with the results of freezing and thawing. Each sample has its own characteristic range in permeability.

The values for average radius of capillaries and number of capillaries per unit area are calculated[10] from the porosity and air-permeability measurements and are poorly correlated with results of freezing and thawing. The average radius ranges from 0.081 to 1.33 μ and, like permeability, varies more from sample to sample than within a given sample. The "number of capillaries (N) per square centimeter" ranges from 503×10^4 to $255{,}000 \times 10.^4$ In general, the higher N's in any given sample are associated with failures by freezing and thawing but each sample has its own limits and these limits vary from sample to sample within a wide range.

Summary and Conclusions

A sample of five bricks was collected from each of ten plants which manufactured bricks from surface clay by the soft-mud process. Each sample represented the temperature range of firing of a kiln. In addition to the customary determination of absorption, saturation coefficient, and strength, measurements were made of porosity and air permeability. All bricks were then subjected to 50 cycles of freezing and thawing, using method B of A.S.T.M. Method C 67.[3] The surviving bricks were then given 125 additional cycles by a method which did not prevent increase in saturation. The data were analyzed to determine what relations, if any, exist between various properties and the results of freezing and thawing.

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[10] The reader is referred to page 719 of Stull and Johnson's paper[7] for a discussion of the assumptions involved in the calculation of these values.

It should be emphasized that the following conclusions do not necessarily apply to types of bricks other than those tested.

1. Fifty cycles of freezing and thawing by method B caused 15 failures out of 50 bricks, whereas 125 additional cycles caused 23 additional failures.

2. Where properties of bricks are related to durability, 50 cycles of freezing and thawing establish a limit for a particular property on one side of which limit no failures take place and on the other side of which limit a random mixture of failures and passes occur.

3. The effect of 175 cycles of freezing and thawing is to leave unchanged the limit on one side of which no failures occur. The range which includes the random mixture of failures and passes is, however, divided by a second limit into a much smaller range where the results continue to be random and into a range where all bricks fail.

4. The following properties of the bricks are sufficiently well correlated with the results of 175 cycles of freezing and thawing to permit limits to be established between the range for no failure, random failure, and complete failure; porosity measured by the gas-expansion method; water absorptions by 1-min. cold immersion, by 24-hr. cold immersion, and by 5-hr. boiling; saturation coefficients measured by porosity (W/V) and by boiling (C/B); and compressive strength.

5. The following pairs of values for the above-listed properties represent the limits for the range of each property within which random failures and passes occur. The figures in parentheses represent the number of specimens within these limits. Porosity (2) 29.8 to 30.9 per cent; water absorption by 1-min. cold immersion (9) 1.9 to 4.2 per cent; by 24-hr. cold immersion (2) 9.7 to 10.9 per cent; by 5-hr. boiling (5) 12.2 to 13.8 per cent; saturation coefficient by porosity, W/V, (8) 0.61 to 0.65; by 5-hr. boiling, C/B, (22) 0.73 to 0.80; and compressive strength (7) 5500 to 6400 psi.

6. The following properties of the bricks are so poorly correlated with durability that limits cannot be established: air permeability, number of capillaries per unit area, average radius of capillaries, and modulus of rupture.

7. In so far as passing a freezing-and-thawing test is accepted as permitting a waiver of the physical properties required for acceptance in the *SW* grade of the A.S.T.M. Specifications for Building Brick (C 62),[4] a more severe test than 50 cycles should be prescribed.

Acknowledgments:

Laboratory measurements and freezing-and-thawing tests were made by J. C. Richmond and W. H. Herschel. Much help in planning the investigation and analyzing the data was provided by R. T. Stull and P. V. Johnson.

AN ANALYSIS OF THE URANYL OXALATE ACTINOMETER AS APPLIED TO ACCELERATED LIGHT AND WEATHERING TESTS

By F. T. Bowditch,[1] C. E. Greider,[1] and C. G. Ollinger[2]

Synopsis

The paper discusses the practical usefulness of the uranyl oxalate actinometer as defined by the Society's Committee D-11 on Rubber Products.[3] Through application of the spectral sensitivity characteristics of uranyl sulfate decomposition to the spectral energy distribution characteristics of typical sources of radiant energy used in accelerated testing, it is shown that this actinometer cannot be used to determine the equivalence of different sources; the spectral energy distributions of two such sources at intensities giving identical uranyl oxalate response are shown to be very widely different.

The usefulness of the uranyl oxalate actinometer in checking the constancy of a source of given spectral composition is pointed out, and practical modifications in the method designed to simplify its use in the average laboratory are discussed.

The increasing use of artificial sources of radiation in the accelerated testing of materials has produced a demand for a simple means for the evaluation of such a source. A procedure designed to accomplish this result, which is included in a Federal specification for the testing of rubber[4] and which has been proposed recently as an A.S.T.M. standard method,[3] is the photochemical decomposition of a solution of uranyl oxalate. This paper presents some of the results obtained in our investigation of this method.

The study of this reaction by Forbes and his associates[5] has provided much of the data necessary for its acceptance as a standard procedure, while its application to the accelerated testing of rubber has been investigated recently by Werkenthin and associates.[6] The proposed method depends on the fact that oxalic acid in the presence of uranium salts is decomposed by ultraviolet radiation and that the amount of this decomposition can be readily determined by the standard analytical procedure of titration with permanganate solution. It thus has the advantage of being a method that can be carried out in any laboratory, with materials and apparatus readily available, and on an absolute basis which may be duplicated by careful users everywhere.

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[1] Research Engineers, Research Laboratories, National Carbon Co., Inc., Cleveland, Ohio.

[2] Manager, Lamp Dept., Carbon Sales Division, National Carbon Co., Inc., Cleveland, Ohio.

[3] Proposed Method of Calibrating a Light Source Used for Accelerating the Deterioration of Rubber, *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 385 (1940).

[4] Rubber Goods: General Specifications (Methods of Physical Tests and Analyses), Federal Specification Zz-R-601a, pp. 24–29, June, 1940.

[5] W. G. Leighton and G. S. Forbes, "Precision Actinometry with Uranyl Oxalate," *Journal*, Am. Chemical Soc., Vol. 52, pp. 3139–3152 (1930).

F. P. Brackett and G. S. Forbes, "Actinometry with Uranyl Oxalate," *Journal*, Am. Chemical Soc., Vol. 55, p. 4459 (1933).

G. S. Forbes and L. J. Heidt, "Optimum Concentrations of Uranyl Oxalate Solutions for Actinometry," *Journal*, Am. Chemical Soc., Vol. 56, pp. 2363–2365 (1934).

[6] T. A. Werkenthin, David Richardson, R. F. Thornley and R. E. Morris, "Equipment for Accelerated Light Aging of Rubber and Means of Evaluation of Ultraviolet Light and Sunlight," *India Rubber World*, Vol. 105, pp. 143–146; 264–268 (1941).

EVALUATION OF PERFORMANCE

An essential step in the evaluation of this method is the determination of the spectral sensitivity of oxalic acid decomposition. This, as calculated from the data of Forbes, is shown in Fig. 1.

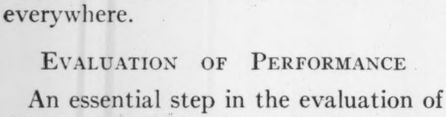

FIG. 1.—Wave Length Sensitivity of Uranyl Oxalate Solution.

Decomposition is produced by radiation throughout the entire range of the ultraviolet, as well as by the violet and blue of the visible spectrum, with a gradually decreasing effectiveness with increasing wave length. The solid curve shown was calculated for a depth of solution of 0.7 cm., since this is the average value found with the cells having a nominal thickness of 1 cm., which we used for this work. As indicated by the dotted curve, and as will be shown later, the spectral sensitivity of the reaction is materially altered by a change in length of the light path through the solution, so that conclusions must be drawn with this variation in mind. The response of the solution is nevertheless in the general range of the spectrum believed to be most effective in accelerated testing, and to this extent it is suited to the intended purpose.

However, a measuring method of this sort can do no more than evaluate the ability of a source to promote a specific photoreaction. The broad fields of accelerated weathering and fading encompass many such reactions, no doubt very widely different in spectral sensitivity, so that the response of any one of them is not a safe measure of the relative behavior of any of the others. This problem is much more complicated than that involved in the measurement of the visual intensities of a number of differently colored light sources, for example. In the latter case there is only the single photochemical reaction of the human eye to be considered, so that a photocell with a suitable filter can be chosen to give accurate results. The present problem might be compared to that of developing a measure of visibility if no two pairs of eyes saw things alike, with some having maximum sensitivity in the blue, some in the green, some in the red and so on in endless variation. A complete specification of the spectral energy distribution of the source would then be the only satisfactory measure of visual intensity, just as it is in the evaluation of the potency of a source for accelerated testing.

Using a given source of constant relative energy distribution, however, a test method such as the uranyl oxalate actinometer can be safely used to determine the absolute intensity of that source from time to time as it may fluctuate with line voltage, with the fogging of glass enclosures, or with other factors which affect the radiant energy at all wave lengths in substantially equal amount. That this instrument, or any other based upon the summation of effects at different wave lengths into a single quantity, cannot be employed for the relative evaluation of sources of widely different spectral characteristics is apparent from the following illustrative example.

The curves of Figs. 2 and 3 give the distribution of radiant energy throughout the ultraviolet, violet, and blue portions of the spectrum for three sources of radiant energy commonly used in accelerated testing. The data for sunlight are those proposed by Moon[7] for use "whenever a single standard solar-radiation curve is needed in engineering calculations for places near sea level." The artificial sources are an open-flame "Sunshine" carbon arc operated at 60 amp., 50-volts a.c. with a filter of "Corex D" glass, and a plain carbon arc enclosed in a Pyrex globe, operating at 16 amp., 138 volts a.c. In Fig. 2 the distances of the two arcs are so adjusted that each gives the same total amount of ultraviolet as natural sunlight.

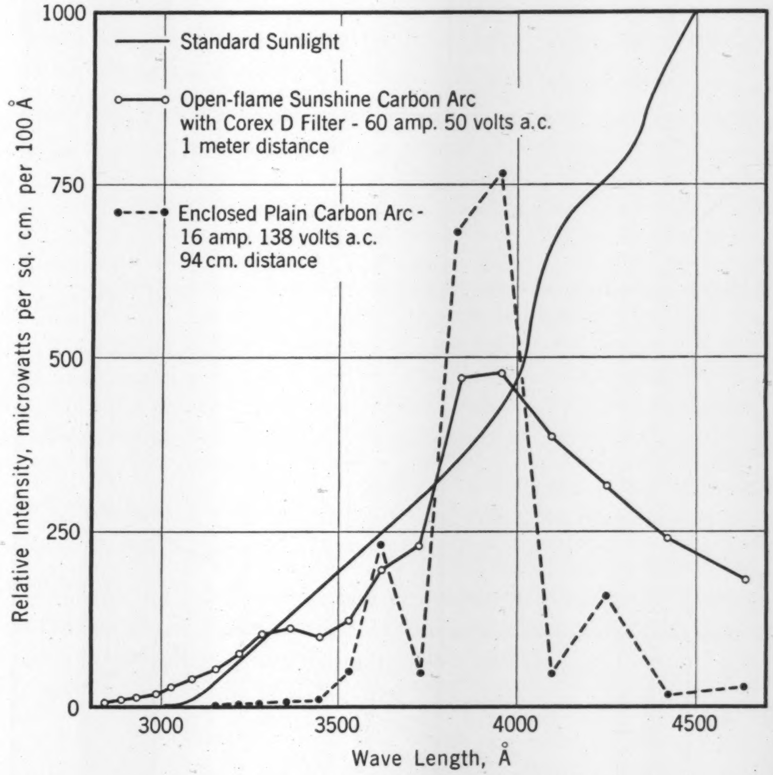

FIG. 2.—Spectral Energy Distribution of Light Sources with Same Total Ultraviolet Intensity.

Figure 3 shows the spectral energy distribution of the same three sources, but this time at distances which give not the same amount of total ultraviolet but the same response with the uranyl oxalate actinometer. In other words, the actinometer says that the intensities of these three sources are identical. Actually, the Sunshine carbon arc has a somewhat higher intensity than sunlight throughout practically the entire ultraviolet, while the enclosed carbon arc has very little energy of wave lengths shorter than 3500 Å and a very much higher intensity than sunlight in the part of the ultraviolet nearest the visible spectrum (3500 to 4000 Å). Assume that a particular photochemical reaction to be accelerated with one of these sources is induced primarily by energy of wave lengths between 3000 and 3500 Å. In this region, the Sunshine arc delivers almost 600, sunshine a little over 400, and the enclosed arc only about 50 microwatts per sq. cm.

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[7] Parry Moon, "Proposed Standard Solar-Radiation Curves for Engineering Use," *Journal*, Franklin Inst., Vol. 230, pp. 583–617 (1940).

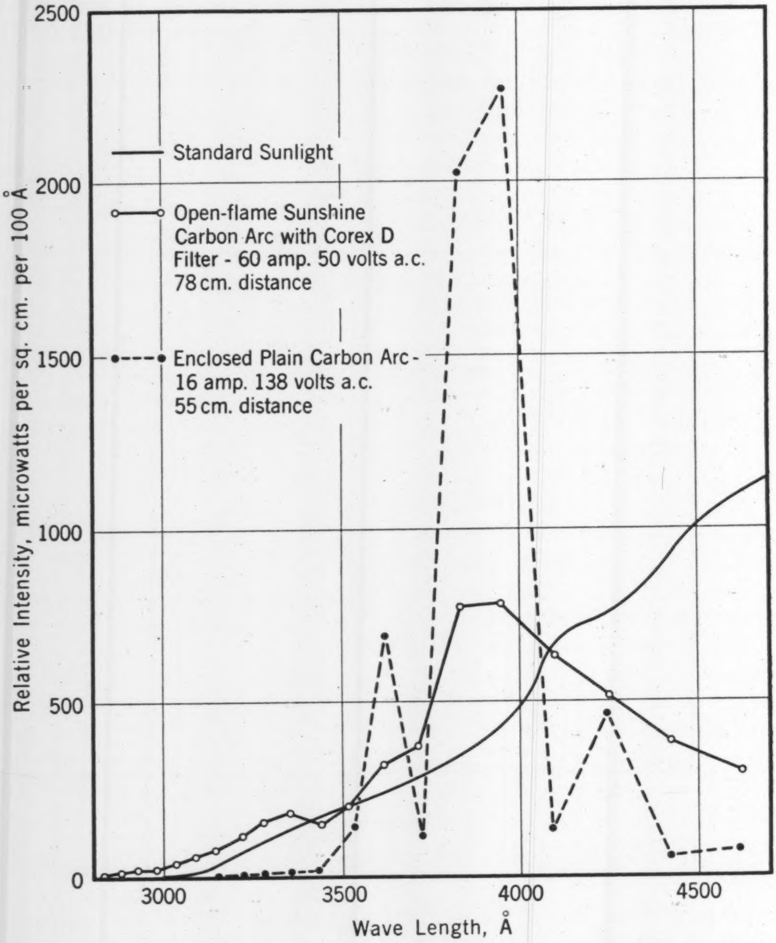

Fig. 3.—Spectral Energy Distribution of Light Sources with Same Uranyl Oxalate Response.

On the other hand, in the near-visual wave length region around 3900 Å, the enclosed arc is almost three times as intense as the Sunshine arc, and over five times as intense as sunshine, so that any photochemical reaction promoted primarily by radiation in this region will be accelerated much faster by this source than by either of the others at the distances chosen.

Thus, even though the uranyl oxalate actinometer can tell no difference between the three sources indicated by Fig. 3, it is obvious that their spectral energy distribution characteristics are in fact quite different and that equivalent test results with a wide variety of photochemical reactions cannot be expected from all three sources. The user, then, should pick the source whose spectral energy distribution most nearly satisfies his requirements, and employ the actinometer solely as a check on the constancy of that source—not in any case to compare its photochemical effectiveness with that of any other source.

SUGGESTED MODIFICATIONS OF PROCEDURE

The procedure proposed by Committee D-11[3] for the application of this method requires the use of two types of quartz cells to hold the solution. The one designated as standard has a solution thickness, measured in the direction of the radiation, of 3 cm., while the corresponding value for the working cell is a little less than 1 cm. Values obtained with the working cell are referred to the standard cell by means of a conversion factor. This procedure is open to the objection that the relative response of the cell to radiation of different wave lengths is not the same for the two thicknesses and therefore the conversion factor will not be a constant one for all sources. Referring to Fig. 1, in the range of cell thicknesses practically employed, the solution absorbs all the radiation of wave lengths shorter than about 3200 Å. In this region, therefore, the amount of decomposition is independent of solution thickness. At longer wave lengths, however, the solution is partially transparent. The amount of radiation absorbed and thus the amount of photochemical reaction therefore increases materially with increasing solution thickness. As shown by the dotted curve, the amount of decomposition caused by energy of wave lengths longer than 3600 Å is more than twice as great for the 3 cm. as for the 1 cm. cell. Since the relative amounts of energy at the different wave lengths vary from one source to another, a factor to convert readings from one cell to the other will hold only for the source with which it was determined. For this reason, it would be preferable either to omit the standard cell altogether, as is done in the similar Federal specification, or to make it of the same nominal thickness as that of the working cell.

The proposed A.S.T.M. method also describes a modification for the measurement of the intensity of ultraviolet of wave lengths shorter than 3350 Å which involves readings taken with and without a filter which absorbs this radiation. The difference between these readings, after correction for reflection losses, is taken as a measure of the energy cut off by this filter. Such a procedure will give a reasonably accurate measure of the quantity desired only if the average of a considerable number of readings is taken with and without the filter. If a single pair of readings is depended on, small and unavoidable variations in the measured intensity may be enormously magnified in the difference, introducing a large measure of uncertainty into the final result. As an illustration, assume that the actinometer readings may vary by ±2 per cent from the true value, and that the difference to be measured is 10 per cent of the actinometer reading without the filter. We then have:

	Without Filter	With Filter	Difference
True value............	100	90	10.0
Maximum variation.....	102	91.8	13.8
Minimum variation.....	98	88.2	6.2

A variation of ±2 per cent in the original readings thus appears as a possible variation of over 2:1 in the final value, giving a grossly inaccurate measure of the true ultraviolet intensity of the source. On account of the time and care required to obtain accurate values by the difference method, its use is therefore undesirable as a routine procedure.

The proposed procedure also requires that the solution after exposure be diluted by no more than about 50 per cent before titration. Since the standard permanganate titration should be carried out at not less than 60 C., it thus becomes necessary to provide special means for heating the sample during titration; otherwise the relatively large volume of cold permanganate solution added would cool the solution below this point. We have found that these departures from standard analytical practice are not necessary. If the solution just before titration is diluted with about 200 ml. of boiling water plus the necessary acid, no further heating is necessary and the accuracy of the test is not materially affected. This requirement of minimum dilution was apparently established in the original work by Forbes and his associates,[5] in which very small volumes of solution were handled. Under the conditions of the present test, its use does not add appreciably to the precision of the results and it might well be omitted.

It is also specified that suitable precautions shall be taken against undue exposure of the solution to room light. Some idea of the care necessary may be obtained from our results of exposure of the solution for several hours at about 3 ft. from a north window on a clear day. This exposure caused the oxalic acid to decompose at the rate of between 4 and 5 per cent per hour. The smallest detectable amount of decomposition, based upon our laboratory experience with the specified solutions in the quantities required, is that which would be produced by an exposure of two to three minutes to strong north light. Thus, while careless exposure to room light should, of course, be avoided, extreme precautions in this regard are unnecessary.

AN EXPERIMENTAL INVESTIGATION OF THE BRITISH STANDARD METHOD FOR THE CRUCIBLE SWELLING TEST FOR COAL

By H. N. Ostborg,[1] H. R. Limbacher,[1] and Ralph A. Sherman[1]

Synopsis

The British Standard Method for the Crucible Swelling Test for Coal, in which a 1-g. sample of coal is heated in a silica crucible over a gas burner at a prescribed rate to form a coke button which is compared to a series of standard outlines for determination of the swelling number, has been investigated by the authors for coals of the United States. The particle size and small variations in the weight of the sample are shown to have little effect on the results. The principal variable that affects the swelling of the coal is the method of application of heat to the crucible. Most of the heat must be applied to the bottom, and, for this reason, a gas burner, as prescribed by the British Method, is found to be more suitable than the types of electric heating that have been tried.

With the exception of one laboratory, good check results were obtained on the same samples distributed to several laboratories.

Data are presented on the relation of the swelling indexes of a number of coals to the performance of these coals on residential stokers. The swelling index is found to be a good indication of the suitability of the coal for performance on these stokers from the standpoint of the coking characteristics. Data are presented from an eastern electric utility that show that the indexes are helpful in the selection of coal for industrial underfeed stokers.

All concerned with the combustion of bituminous coal have observed the varying degrees of expansion or swelling exhibited by different coals, and many have voiced the need for a simple, easily duplicated, standard test for the measurement of these properties that the performance of the coals in fuel beds may be predicted. Although the swelling property of coal has been defined in many ways, for the purpose of this paper, the definition agreed upon by Subcommittee XV on Plasticity and Swelling of Coal of the Society's Committee D-5 on Coal and Coke[2] will suffice:

"*Free Swelling* is the property of certain coals when heated, without restraining influence, to expand freely in volume, as in the volatile matter test or when burned as a fuel.

"*Expansion* is the property of certain coals to change in volume when carbonized, with restraining influence, as in a coke oven."

Thus a distinction is made between the changes taking place when coal is burned in a fuel bed or carbonized in a

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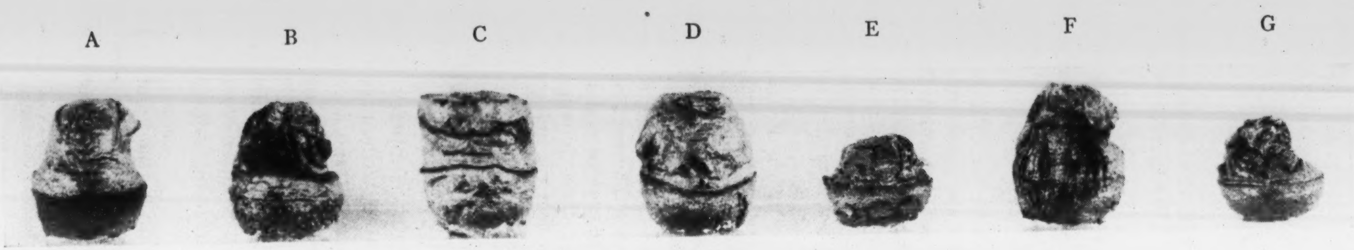
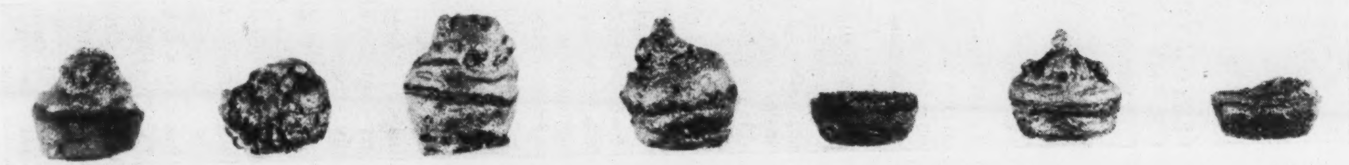
[1] Research Engineer, Research Engineer, and Supervisor of Fuels Division, respectively, Battelle Memorial Institute, Columbus, Ohio.

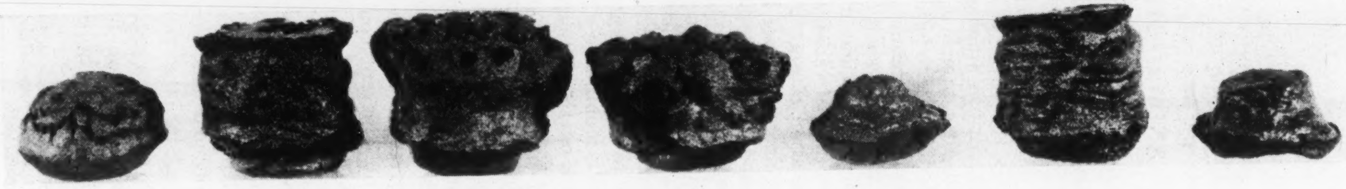
[2] Minutes of meeting of Subcommittee XV on Plasticity and Swelling of Coal of the Society's Committee D-5, held June 28, 1937.



Residue from volatile matter test, Meker burner method, 10-cc. platinum crucible.



Residue from volatile matter test, electric furnace method, 10-cc. platinum crucible.



Residue from British Standard Method for the Crucible Swelling Test for Coal.

FIG. 1.—Coke Buttons Obtained by Heating in Standard Electric Furnace for Volatile Matter Determination, by Heating over a Meker Burner and Using the British Standard Method for the Crucible Swelling Test for Coal.

coke oven, indicating the need of more than one type of test.

Numerous test methods and apparatus have been developed to determine the expansion and swelling properties of coal. Among the tests for the expanding properties, some of which are described by Malleis,[3] are: (1) Altieri method, (2) Bethlehem method, (3) U. S. Bureau of Mines ovens, and (4) Pennsylvania State College Laboratory scale oven. For the measurement of the swelling properties, the following have been developed: (1) Agglutinating test, (2) Examination and determination of volume of coke button from standard volatile-matter test (Fuels Research Laboratory test), and (3) the British Standard Method for the Crucible Swelling Test for Coal.[4]

As described by Malleis,[3] the Agglutinating Test in which the crushing strength of coke buttons formed by carbonization of mixtures of coal and an inert material as sand or silicon carbide seems to have little value as a reliable, readily duplicated index of the probable coking or caking properties of coal. Malleis showed, however, that it has been found to be of value for special investigations such as detecting deterioration of coking properties due to storage. The use of a crucible swelling test for the determination of the free swelling property of a coal is by no means a recent innovation, as both Malleis[3] and Rose and Sebastian[5] indicated that the coke button resulting from the standard volatile-matter test is an indication of the coking property of the coal. Both investigators referred to the coke buttons as being "swollen" buttons. However, it has been shown that the size of the button from this test is quite dependent upon the type of heating employed.[6] Figure 1[7] illustrates this point. Table I lists the coals from which these coke buttons were made.

The electric furnace method resulted in smaller buttons than the gas flame method. This is particularly evident in a comparison of the coke buttons made by the two methods for the low-volatile Pocahontas No. 3 coal. The British Standard Method gave larger buttons than those formed by either of the two standard volatile determination methods.

TABLE I.—COALS USED IN MAKING COKE BUTTONS ILLUSTRATED IN FIG. 1.

Coke Button	Coal Seam	Volatile Matter, Dry Basis, per cent
A.....	Lower Kittanning	17.0
B.....	Pittsburgh	35.6
C.....	Beckley	20.5
D.....	Pocahontas No. 3	19.0
E.....	Illinois No. 6	36.1
F.....	Sewell	26.3
G.....	Kentucky High Splint	39.0

The fact that the British Crucible Swelling Test had been adopted by the British Standards Institution led to its selection for trial and for further investigation after the trials showed it to hold promise of being a quick, simple test with a high order of duplicability and with significance in combustion of coal in fuel beds.

This paper describes the method with such modifications as have been required, and discusses, (1) the effect of the particle size of the sample, (2) the effect of variations in the weight of sample, (3) the effect of variations in the rate of

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[3] O. O. Malleis, "Laboratory Tests Relating to Caking, Plastic, Gas- and Coke-Making Properties of Bituminous Coals," *Proceedings*, Am. Soc. Testing Mats., Vol. 37, Part II, p. 402 (1937).

[4] British Standard Method for the Crucible Swelling Test for Coal, British Standards Institution, No. 804–1938, July, 1938.

[5] H. J. Rose and J. S. Sebastion, "Changes in Properties of Coking Coals Due to Moderate Oxidation During Storage," *Transactions*, Coal Division, Am. Inst. Mining and Metallurgical Engrs., Vol. 88, p. 556 (1930).

[6] Discussion by Ralph A. Sherman of paper by O. O. Malleis, Laboratory Tests Relating to Caking, Plastic, Gas- and Coke-Making Properties of Bituminous Coals," *Proceedings*, Am. Soc. Testing Mats., Vol. 37, Part II, p. 417 (1937).

[7] W. A. Selvig, "Experience with Results of Tests Using British Standard Method for the Crucible Swelling Test for Coals," Private communication to Ralph A. Sherman, October 24, 1939.

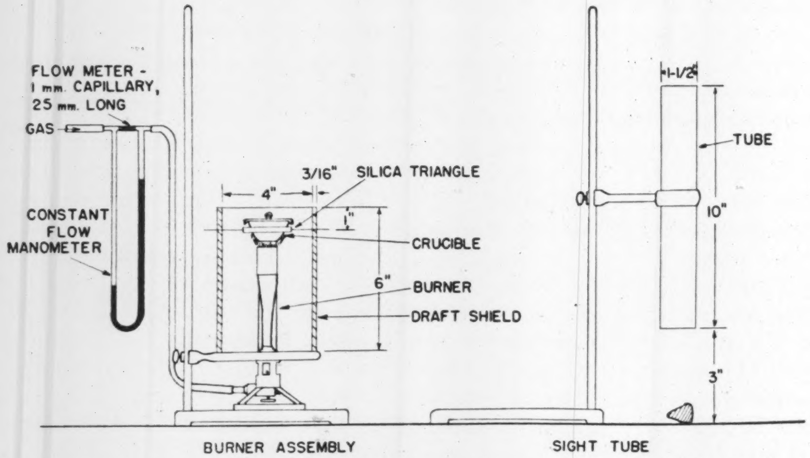
FLOW METER -
1 mm. CAPILLARY,
25 mm. LONG
GAS
CONSTANT
FLOW
MANOMETER
3/16"
SILICA TRIANGLE
4"
1"
CRUCIBLE
6"
BURNER
DRAFT SHIELD
1-1/2"
TUBE
10"
3"
BURNER ASSEMBLY
SIGHT TUBE

FIG. 2.—Details of Burner Assembly and Sight Tube Construction for British Standard Method for the Crucible Swelling Test for Coal.

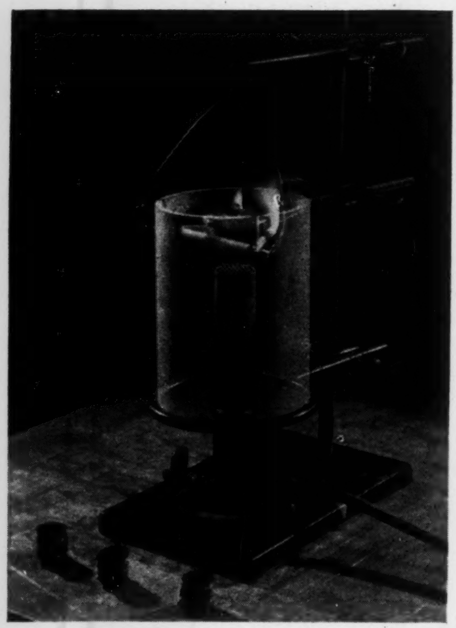

FIG. 3.—Photograph of Burner Assembly, showing Crucible in Place, for British Standard Method for the Crucible Swelling Test for Coal.

heating, (4) the order of duplicability of results by different laboratories, (5) some trials of electric heating to replace gas heating, and (6) the significance of the swelling index with relation to the performance of coals on residential and industrial underfeed stokers.

TEST EQUIPMENT

The test method as described by the British Standard consists essentially of heating 1 g. of coal, in a covered silica crucible in the flame of a gas burner so adjusted as to give a temperature of 800 ± 10 C., in $1\frac{1}{2}$ min. and 820 ± 5 C., in $2\frac{1}{2}$ min. The heating is continued for not less than $2\frac{1}{2}$ min. or until the flame of the burning volatile matter has disappeared. The coke button obtained is compared with a series of standard outlines, numbered 1 to 9 in steps of half numbers and given a value corresponding to that of the nearest outline.

Figures 2 and 3 show the test assembly. The crucible is made of trans-

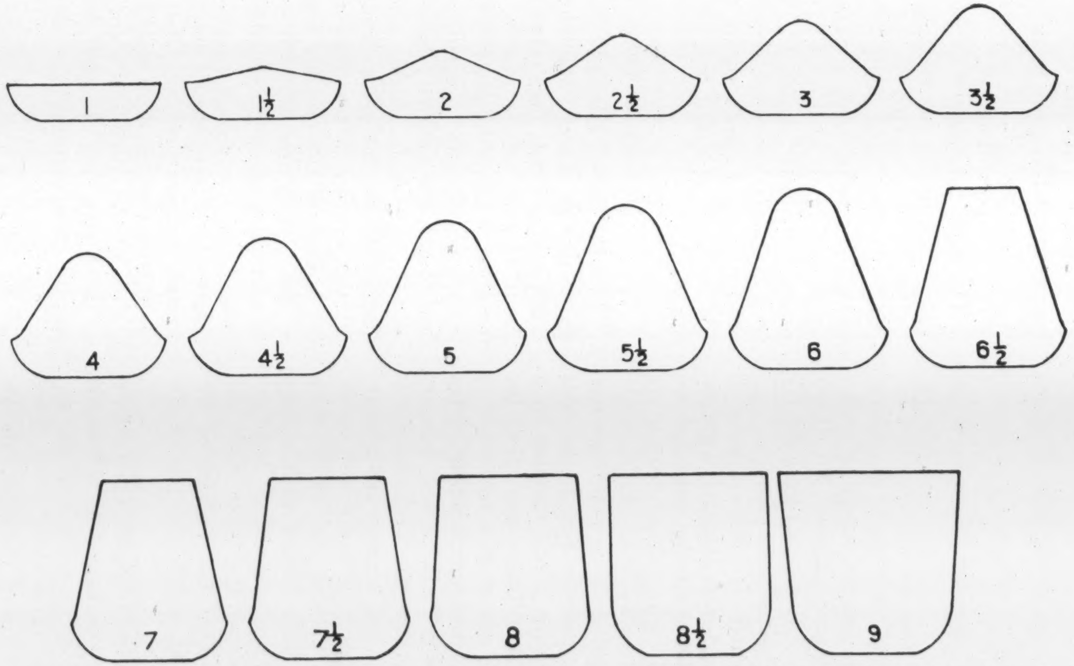
1
1½
2
2½
3
3½
4
4½
5
5½
6
6½
7
7½
8
8½
9

FIG. 4.—Full Scale Standard Profiles and Corresponding Swelling Numbers for British Standard Swelling Test for Coal.

lucent silica, squat shape, with a silica, ring-handled lid. The British Standard lists the following specifications for the crucible:

1. *Weight*—not more than 12.75 or less than 11.0 g.
2. *External height*—26 ± 0.5 mm.
3. *External diameter at top*—41 ± 0.75 mm.
4. *Internal diameter at base*—not less than 11 mm.
5. *Capacity*—approximately 17 ml.

Low form, glazed Vitreosil, No. 21246, or Amersil, No. CO, crucibles meet the above specifications quite closely.

The triangular crucible support is made of 6 to 6.5-mm. external diameter, translucent silica tubing mounted on chrome-nickel wire. The length of the side is 63 to 64 mm.; the diameter of the inscribed circle is approximately 32 mm. As shown in Fig. 3, the ends of the triangle are joined together by a loop of wire to facilitate removal of the crucible at the end of each test.

The original test specifications called for the use of a Teclu burner, $\frac{1}{2}$ in. in diameter. Because the Teclu burner is not available in this country and because a burner of that type is not well suited for use with natural gas where it is available, a Fisher burner with a large grid, 1 $\frac{3}{16}$-in. external diameter, has been used. The burner and draft shield are so positioned that the bottom of the crucible is $\frac{3}{8}$ in. above the top of the burner.

The draft shield is made from asbestos cement piping 6 in. long, 4 in. in internal diameter, and $\frac{3}{16}$ in. thick. At the top the piping has three slots, 1 in. deep, in which the wires of the silica triangle rest. This assures placing the crucible in the same position for succeeding tests.

A capillary flow meter with water manometer is placed in the gas line before the burner as a guide to the control of the rate of gas flow. This has proved to be a time-saving device, as it eliminates check determinations of the crucible temperature when many tests are to be run in one day. The temperature determinations are made by the use of a thermocouple of chromel-alumel wire and a potentiometer.

The sight tube shown at the right of Fig. 2 is a device for viewing the coke buttons which eliminates the effect of parallax. The tube itself is a simple affair and may be made of either glass or metal tubing 10 in. long and $1\frac{1}{2}$ in. in internal diameter supported vertically on a ring stand above the sheet of standard outlines. Figure 4 shows, full scale, the outlines specified by the British Standard.

DETAILS OF TEST

With the burner assembly arranged as shown in Figs. 2 and 3 and with a blank crucible in position, the burner is lighted about 15 min. before making a determination to allow the draft shield to rise to an equilibrium temperature. The flow of gas, the relative positions of the burner, the draft shield, and consequently the crucible are so adjusted that the temperature of the inner surface of the bottom of the crucible reaches 800 ± 10 C., in $1\frac{1}{2}$ min. and 820 ± 5 C., in $2\frac{1}{2}$ min. after placing a crucible in position.

Using the Fisher burner described, the desired temperatures may be attained by so adjusting the draft shield that the bottom of the crucible is approximately $\frac{3}{8}$ in. above the burner grid. The crucible temperatures are determined by inserting the thermocouple through a pierced lid until the unprotected junction of the couple is in contact with the base of the crucible. The end of the thermocouple should be in the form of a flattened loop and the junction and a portion of each wire should rest on the bottom of the crucible during the temperature measurements. Once the conditions for attaining the correct heating have been ascertained, it is well to fix permanently the position of the draft shield and burner, assuring ease of starting operations for succeeding tests.

One gram of freshly ground 0–60 mesh coal is next weighed in a cold crucible, and the crucible is lightly tapped 12 times on the bench, rotating it between taps, to level the surface of the coal. The crucible is then covered with a lid and placed upright in the silica triangle supported in the draft shield. The crucible and triangle may be placed into the draft shield together, by the use of the wire frame connecting the three legs of the triangle over the crucible. This makes it unnecessary to turn off the burner between each heat.

The covered crucible is heated in the gas flame for such a time as is required for the flame of the burning volatile matter to die out, and in any case for not less than 2½ min. The button is carefully removed, and the carbon residue remaining in the crucible is removed by ignition.

Four buttons are made for each sample of coal tested. Thus, at least four crucibles and lids are required with one extra lid to be used during the temperature determinations.

METHOD OF REPORTING RESULTS

When four coke buttons have been made from the sample of coal being tested, each button is viewed through the sight tube and compared to the series of standard profiles. The drawing with which the button is to be compared is placed exactly in the center of the field of vision from the top of the tube. The button is rotated around its axis until the maximum cross-section area is in line with the drawing and viewed with one eye placed immediately over the top of the tube.

The swelling number is the number inscribed in the outline that the largest profile of the button most nearly matches, and the average swelling number of the series, expressed to the nearest ½ unit, is reported. The practice of the authors has been to disregard a button if it deviates more than 1 unit from the other three, and to make a new determination.

EFFECT OF PARTICLE SIZE OF SAMPLE

The British Standard specifies that the particle size of the coal sample shall be such that it will pass a B.S. 72 mesh test sieve. This corresponds to a U.S.S. No. 70 sieve. Because the analytical tests for coal in the United States are based on a sample prepared to pass a U.S.S. No. 60 sieve,[8] the data determined by the authors were obtained by using samples crushed to the latter size. Several of the collaborating laboratories used a U.S.S. No. 70 sieve, but no appreciable difference in the results was noted because of this.

TABLE II.—BRITISH STANDARD SWELLING NUMBERS FOR FOUR COALS USING MINUS 60 MESH AND PULVERIZED SAMPLES.

Coal	British Standard Swelling Number	
	Minus 60 mesh	Pulverized
Pocahontas No. 5	9	9
Pocahontas No. 3	5½	5½
B seam	6	6
C prime	9	9

A series of swelling tests was made on coals that were prepared for pulverized firing, that is, to approximately 70 per cent less than 200 mesh. It was much more convenient to obtain a sample of the pulverized coal as it was being pulverized, than to crush down the larger sample to minus 60 mesh. Before adopting this practice, however, duplicate swelling determinations were made on four different coals using both the minus 60 mesh and pulverized samples. Table II lists the swelling numbers obtained for the four coals.

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[8] Standard Method of Laboratory Sampling and Analysis of Coal and Coke, (D 271 – 40), 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 5.

The exact agreement indicates that crushing to minus 60 mesh or finer is satisfactory and that the exact size is not particularily critical for the test.

TABLE III.—RESULTS OF BRITISH STANDARD SWELLING TESTS ON FOUR COALS USING TWO SIZES OF A MEKER BURNER,[a] AND ONE FISHER BURNER

Coal	British Standard Swelling Number		
	Bureau of Mines Meker Burner[a]		Battelle Fisher Burner
	1-in. grid	$1\frac{3}{16}$-in. grid	$1\frac{3}{16}$-in. grid
Indiana No. 6	1	1	$1\frac{1}{2}$
Fulton	7	9	9
Island creek	$4\frac{1}{2}$	7	7
Pittsburgh	3	5	6

[a] W. A. Selvig, "Experience with Results of Tests Using British Standard Method for the Crucible Swelling Test for Coal," Private communication to R. A. Sherman, October 11, 1939.

EFFECT OF SMALL VARIATIONS IN THE WEIGHT OF SAMPLE

To determine the accuracy required in weighing the 1-g. test sample, two series of tests were run. In the first test the coal was weighed accurately to 0.1 mg. on an analytical balance, and in the second series the sample was weighed to 0.1 g. on a laboratory three-beam platform balance. The size of the coke buttons made by these two procedures was the same. This shows that extreme accuracy is not needed in weighing out the sample, and that the normal laboratory platform balance, graduated in 0.1 g., is sufficiently accurate for the test.

EFFECT OF RATE OF HEATING

Because of the demonstrated difference between buttons made by the electric-furnace and gas-burner methods for volatile-matter determinations, the rate of heating of the sample would be expected to be an important factor governing the size of the buttons. A number of items have bearing upon the proper heating rate, such as: (1) type of gas, manufactured or natural; (2) position, size, and type of the burner; (3) dimensions and position of the crucible in relation to the burner, and (4) thickness of the draft shield.

Effect of Supply of Gas:

Two of the seven laboratories that exchanged coal samples to test the order of duplication of results used manufactured gas, whereas the other five used natural gas. Of the two laboratories using manufactured gas, one obtained results that checked well with the others, but the second obtained results that were considerably lower than the data by the other laboratories. The reason for this discrepancy is not known, although it is believed differences in burner design and in flame characteristics of the two gases may be responsible.

Effect of Variations in the Gas Burner:

Table III shows the difference in results on four coals because of the difference in the size of the burner.

Using the Meker burner having a grid diameter of 1 in., the bottom of the crucible was $\frac{3}{8}$ in. above the top of the grid, and using the Meker burner having a grid diameter of $1\frac{3}{16}$ in., the bottom of the crucible was $\frac{3}{4}$ in. above the top of the grid. The results obtained with the larger sized Meker burner were very similar to the data obtained with the Fisher burner, as the burning characteristics of the two burners, with the same grid size, are quite similar, although the distance between the top of the Fisher burner grid and the bottom of the crucible was only ⅜ in. With the longer and broader flame, the system had a larger heat capacity, so the heat input to the coal differs considerably for the two different sized burners. To eliminate this source of error all laboratories should use the same size burner with the same distance between the burner top and the bottom of the crucible, and consequently the same length of flame travel.

The burner should be so positioned that the crucible is directly over the center of the burner grid. This will assure a maximum of heating from the bottom and a minimum of side heating. Heating principally from the bottom of the crucible allows the coke button to swell up and out, whereas too much side heating cokes the coal quickly at the sides and top; this prevents free swelling of the coal.

TABLE IV.—RESULTS OF BRITISH STANDARD SWELLING TESTS RUN ON PITTSBURGH COAL, USING VARIOUS CRUCIBLE TEMPERATURES.

Sample	Temperatures, deg. Cent.				Swelling Number
	Crucible Temperature		Deviation from Standard		
	1½ min.	2½ min.	1½ min.	2½ min.	
No. 1	757	781	−43	−39	6
No. 2	778	800	−22	−20	7
No. 3	793	810	−7	−10	6½
No. 4	805	817	+5	−3	7
No. 5	812	830	+12	+10	7
No. 6	819	836	+19	+16	6½

Effect of Variations in Temperature:

A series of tests was run on Pittsburgh seam coal to determine the critical nature of the stipulated crucible temperatures. The gas flow was held constant and the temperature was varied by raising and lowering the draft shield and crucible. Table IV lists the swelling numbers obtained with the various crucible temperatures. All the buttons were made in the same crucible to eliminate this variable.

Thus, within wide limits of temperature, no radical difference in button size was observed. This comparatively wide range of safe temperature would seem to eliminate the effect of small variations in crucible dimensions. This does not mean, however, that the established test procedure should be markedly departed from. Because most of the heat is supplied to the bottom of the crucible, the thickness of the crucible bottom should be standardized and held within close limits.

TABLE V.—RESULTS OF BRITISH STANDARD SWELLING TESTS ON THREE COALS, USING THE GAS FLAME AND ELECTRIC FURNACE METHODS

Coal	British Standard Swelling Number		
	Gas Flame Method	Electric Furnace Method	
		Without Adapter	With Adapter
A	2	2	2
B	5	6	4½
C	9	8	8 to 8½

The thickness of the draft shield has a direct effect upon the crucible temperature, but only while the shield is warming up. Using first a draft shield with a wall thickness of ½ in., it was found that a 30-min. period must elapse before stable crucible temperatures were attained. The wall thickness was then reduced to $\frac{3}{16}$ in., and the length of the warm-up period was found to have been reduced to 10 min.

Trials of Electric Heating:

The greatest potential source of error in the determination is the rate of heating of the crucible. Differences in burner design and type of gas used introduce the greatest variations. Presumably, such variables could be eliminated and the test method better standardized by the use of electric heating.

A number of types of electric heaters were tried: namely, cones, plates, and tube furnaces. The most successful of these was an electric tube furnace constructed of a ceramic core $2\frac{1}{2}$ in. in internal diameter and $5\frac{7}{8}$ in. high, wound with No. 19 gage chromel-A resistance wire. The top 1 in. was wound with three turns per inch and the rest of the core was wound with six turns per inch. This was done to insure that most of the heat was being supplied to the bottom of the crucible. This was not entirely successful, as in order to reach the de-

TABLE VI.—RESULTS OF BRITISH STANDARD SWELLING TESTS FOR VARIOUS LABORATORIES ON THE SAME COAL SAMPLES.

Sample		British Standard Swelling Number			
		Laboratory No. 1	Laboratory No. 5	Laboratory No. 6	Laboratory No. 7
Indiana No. 6	6M by 1¼ in.	1½	1	1	1 to 1½
Pittsburgh	0 by ¾ in.	6	5	5	4 to 4½
Island Creek	⅜ by 1¼ in.	7	7	7	5½
Fulton	0 by 1½ in.	9	9	10½	7 to 7½
C Prime		9	..	8½	5½ to 6
E Seam		9	..	11	5
Pocahontas No. 3		6½	..	6	3½ to 4
Pocahontas No. 3		8	..	8	4½

Sample		Laboratory No. 1	Laboratory No. 2	Laboratory No. 3	Laboratory No. 4
Floater	Broad Top District	9	8½	..	..
Dull Band	Broad Top District	7½	8	..	..
Bright Band	Broad Top District	9	9	..	..
Hubbard	0 by ⅜ in.	9	8	..	..
Rockhill		9	8½	..	..
Alabama	Mary Lee Mine	7½	8½	..	..
Barnes No. 15		9	9	..	..
Kentucky		5	6	..	..
Illinois No. 5		2	2½	2½	..
Kentucky No. 14		4	3½	3½	..
Chilton		6½	6½	6½	..
Sewell		9	9	9	..
Magee	1¼ by 2 in.	8½	..	8½	..
Magee	0 by 1¼ in.	9	..	8½	..
Hutchinson	½ by 1¼ in.	8½	..	9	..
Hutchinson	0 by ½ in.	8½	..	9	..
Hocking No. 6	⅜ by 1¼ in.	1½	..	..	1
Wallins	0 by 1¼ in.	3	..	..	3
Island Creek	⅜ by 1¼ in.	6½	..	..	6½
Powellton		9	..	..	8½
Illinois No. 6	¼ by ¾ in. from 2 by 4 in.	4½	..	..	4½
Illinois No. 6	0 by ¼ in. from 2 by 4 in.	4	..	..	5
Illinois No. 6	¼ by ¾ in. from 1¼ by 2 in.	4	..	..	5
Illinois No. 6	0 by ¼ in. from 1¼ by 2 in.	4	..	..	4½
Illinois No. 6	¼ by ¾ in. from ¾ by 1¼ in.	4	..	..	4½
Illinois No. 6	0 by ¼ in. from ¾ by 1¼ in.	4½	..	..	4½
Illinois No. 6	¼ by ¾ in.	4½	..	..	4½
Illinois No. 6	48M by ¼ in.	4½	..	..	4½

Sample	Laboratory No. 3	Laboratory No. 6	Laboratory No. 7	
Lower Kittanning	..	9	4½	
B Seam	..	9+	6½ to 7	
Pittsburgh	..	9+	6½	
Pocahontas No. 3	..	9	5	
Eccles	8½ to 9	..	5 to 5½	
Crozer (Upland)	9	..	5 to 5½	
McCullough	8 to 8½	..	7½ to 8	
Magee	8½ to 9	..	7½ to 8	

sired test temperatures the crucible had to be placed entirely within the furnace, which resulted in excessive side heating. The resulting coke buttons were similar to those formed by the gas-flame method, but the excessive side heating pinched the button in at the top, giving a "bottle-shaped" effect. Much heat was lost from the top of the furnace during the test, and a period of 20 min. was required between each determination to bring the furnace back to the proper temperature.

An adapter of insulating refractory was so fitted into the top of the furnace that its internal diameter closely approximated the external diameter of the crucible. This effectively reduced the loss of heat from the end of the furnace as the time interval between tests was reduced from 20 to 2 or 3 min.

Table V gives comparative data for three coals for which coke buttons were made by the gas-flame method and by the electric tube furnace, with and without the adapter. The excess side heating of the crucible was eliminated by use of the adapter and the resulting coke buttoms were essentially the same size as those determined by the gas-flame method. These data indicate promise of standardization of the test by the use of an electric furnace, but the gas-burner method has been shown to give such good results that further attempts to develop an electric furnace were not considered warranted.

Other Methods for Determination of Size of Button:

Some experimenters have tried other methods of measuring the size of the coke button, such as volume and cross-sectional area determinations. It is believed, however, that the size designation is not critical and that the standard profile method is satisfactory.

POSSIBILITY OF DUPLICATION OF RESULTS

In one laboratory, no difficulty has been found in agreement of duplicate determinations. To learn the possibility of agreement of different laboratories, several sets of samples have been distributed to a number of laboratories. Table VI gives the data obtained.

Swelling indexes as given by laboratory No. 6 are determined by cross-section area rather than by the standard profile method. Consequently, they report some values greater than 9. On samples having indexes of 8 or less the agreement with other laboratories is good.

The only laboratory that gave results materially different from the others was No. 7. Although an attempt was made by exchange of correspondence to learn the reason for the difference, no definite conclusion was reached. That laboratory used manufactured gas with a Precision Hy-Temperature burner, with the flame grid replaced with the grid from a Fisher burner. It is suspected that a difference in the distribution of the heat to the bottom and side of the crucible resulted, but the fact that laboratory No. 6 also used manufactured gas and checked the results of other laboratories shows that the type of gas was not the determining factor.

In general, the results show that there should be no difficulty in checking determinations among laboratories, if each is careful to check frequently the rate of heating obtained in the crucible by means of a thermocouple as prescribed in the section on Details of Test, and to use the same type and size of burner.

RELATION OF SWELLING INDEX AND PERFORMANCE OF COALS WHEN BURNED IN A STOKER

The coking property of a coal is one of the most important characteristics that govern the performance when burned in an underfeed stoker. In a stoker fuel bed, conditions are so favorable for coking because of the rapid rate of heating of the coal, that coke is formed even with coals normally considered noncoking. In residential stokers excessive coke formations cause deep fuel beds which give trouble in removal of clinker. They result in a slow rate of pick up which, in extreme cases, results in extinction of the fire.[9] In industrial stokers, excessive coke formations cause improper distribution of the coal and air, lead to bad clinkering, smoke, and slow response of the fire.

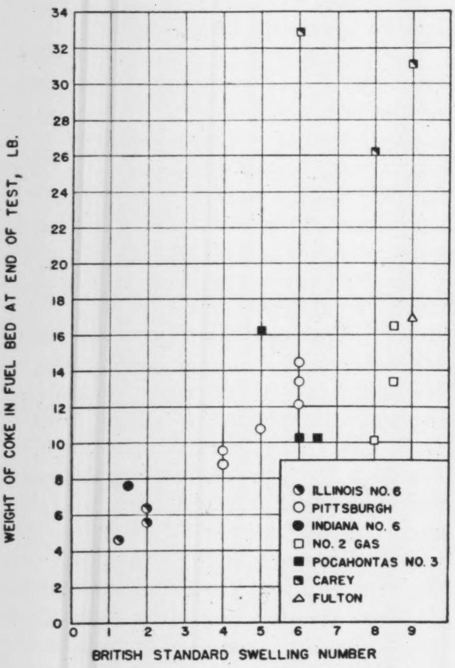

FIG. 5.—Relation of British Standard Swelling Number to Coke Formation in Fuel Bed of Small Underfeed Stoker, Excess Air 35 to 60 per cent.

In an attempt to express the coking in small stoker fuel beds numerically, Sherman, Kaiser, and Limbacher[10] have weighed the coke larger than 1 in. that was left in the fuel bed when it was quenched after uniform burning conditions had been established.

Figure 5 shows the relation of the British Swelling Index of a number of coals to the coking on the stoker as expressed by the weight of coke in the fuel bed. With the exception of the three coals from the Carey seam, there is a fairly good relation between the two values. The relation is partly obscured by the fact that the excess air which also affects the weight of coke in the bed could not be held constant with all the coals. Experience in the laboratory has shown that coals which have a swelling index of six, or less, will cause no difficulty from coke formations in the fuel bed of the small underfeed stoker. Those having indexes of 8 to 9 may or may not cause difficulty but are suspect.

The British Swelling Test has been used by an eastern electric utility, as a guide in the selection of coals for their industrial sized stokers. Table VII lists the swelling indexes obtained for various coals with notes on general burning operation in these industrial units.

An unsatisfactory coal did not stay on the grates and seemed to blow and fluff up more than normally. The fire was not level and compact, and the burning lanes were serpentine instead of straight and at intervals seemed to break over from one tuyere to the other. The maximum output of the boiler dropped about 22 per cent and the fire was very unstable and hard to regulate without producing smoke. All these difficulties were attributed to unsatisfactory coking tendencies. The above data represent only a few coals, for the most part from the Pocahontas No. 3 seam, all of slack size.

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[9] Ralph A. Sherman, "The Evaluation of Coal for Use in Domestic Stokers," *Bulletin*, University of Illinois, Vol. 37, pp. 39–57 (1939).

[10] Ralph A. Sherman, E. R. Kaiser, and H. R. Limbacher, "The Relation of the Size of Bituminous Coals to Their Performance on Small Underfeed Stokers," *Technical Report No. 1*—Part II, Bituminous Coal Research, Inc., July, 1937.

The upper limit of satisfactory operation appeared to be at an index of about 5½. However, the laboratory that made these determinations was the one that obtained results generally lower than the other laboratories that exchanged samples. On the basis of the values obtained by the other laboratories, the upper limit of satisfactory operation would probably have been raised to an index of about 7. Nevertheless, the method proved valuable in evaluation of coals for use on the stokers.

TABLE VII.—RELATION OF BRITISH STANDARD SWELLING INDEX WITH BURNING PERFORMANCE OF VARIOUS COALS ON LARGE UNDERFEED STOKERS.

Sample	Volatile Matter, per cent	Swelling Index	Remarks
No. 1343	17.1	3½	Satisfactory. Burned mixed with another similar coal.
No. 1312	19.0	4	Satisfactory
No. 966	18.0	4½ to 5	Satisfactory
No. 1342	23.0	6½	Unsatisfactory. Burned mixed with another coal.
No. 956	22.1	7	Unsatisfactory
No. 1091	21.8	8½ to 9	Very unsatisfactory
No. 500	22.3	9	No trouble at light loads
No. 1337	16.6	3 to 3½	Best results
No. 1336	20.4	5 to 5½	No experience
No. 1340	20.6	6	Not satisfactory
No. 498	22.3	8	No trouble at light loads
No. 1314	18.7	4½	Satisfactory
No. 1341	22.7	6½	No experience
No. 1311	19.1	4	Satisfactory
No. 1313	18.5	4	Satisfactory
No. 1315	19.1	4	Satisfactory
No. 1338	17.5	4 to 4½	Satisfactory
No. 1354 to 1358	18.5	4	Satisfactory
No. 1369 to 1373	18.9	5	Fair
No. 1378 to 1381	17.3	5	Very satisfactory. Coal seemed coarser.
No. 1382	17.7	3½	Satisfactory. Burned in combination with other coal.
No. 1383	20.6	4½	Unsatisfactory. Burned in combination with other coal.
No. 1385	18.8	5½	Unsatisfactory when burned with other satisfactory coals.

TABLE VIII.—BRITISH STANDARD SWELLING NUMBER OF VARIOUS COALS.

Seam	State	County	Size	Swelling Number
B	Pennsylvania	Clearfield	¾ in. slack	9
B	Pennsylvania	Somerset	Slack	6
Carey	Virginia	Buchanan	Pea	8
Chilton	W. Virginia	Logan	⅜ by ¾ in.	7
C Prime	Pennsylvania	Somerset	¾ in. slack	8½
Elkhorn	Kentucky	Letcher	Furnace lump	7
Illinois No. 2	Illinois	Grundy	¼ by 1¼ in.	3
Illinois No. 2	Illinois	Grundy	0 by 1½ in.	1½
Illinois No. 5	Illinois	Fulton	5/16 by ¾ in.	3
Illinois No. 6	Illinois	Franklin	0 by ¾ in.	1
Illinois No. 6	Illinois	Perry	¼ by ¾ in.	4½
Kentucky No. 14	Kentucky	Hopkins	1¼ by 2 in.	4½
Miller	Pennsylvania	Cambria	½ in. slack	9
Miller's creek	Kentucky	Johnson	0 by 1 in.	1½
No. 2 gas	W. Virginia	Fayette	10 mesh by 1 in.	8½
Pittsburgh	W. Virginia	Putnam	10 mesh by 1 in.	4
Pocahontas No. 3	W. Virginia	McDowell	0 by 1¼ in.	5
Pocahontas No. 3	W. Virginia	McDowell	1¼ in. nut and slack	8½
Pocahontas No. 4	W. Virginia	McDowell	Stoker	6
Pocahontas No. 5	W. Virginia	McDowell	Slack	9
Upper Freeport	Pennsylvania	Armstrong	¾ in. slack	9
Wallins	Kentucky	Harlan	¼ by 1¼ in.	3

SWELLING INDEXES FOR VARIOUS COALS

Table VIII gives the swelling indexes for a number of coals of various original sizes that have been measured at Battelle during the course of this investigation in addition to those given in Table VI.

CONCLUSIONS

The British Standard Swelling Test is an advantageous method of measuring the "free swelling" property of a coal, inasmuch as it is a simple test requiring no complicated apparatus and because the results are easily duplicated in the same and different laboratories. The resulting coke button is large enough that discrimination may be made between borderline coals.

Experimental data obtained for four coals show that the particle size of the sample was not critical. The weight of the 1-g. sample is shown to be sufficiently accurate for the test if weighed to 0.1 g.

The method of heating the crucible is the most important factor of the test method, and care should be taken that all laboratories use the same type of burner with the crucible a set distance above the burner grid in order to assure good duplication of results.

Exchange of coal samples between seven laboratories show, with the exception of one laboratory, that the determinations should check within $\pm\frac{1}{2}$ number. A fairly satisfactory relation was found between the swelling index of coals and the weight of coke formation in small underfeed stokers. One electric utility has found the method of value to predict the performance of coals on large underfeed stokers.

Other laboratories are urged to adopt the method as a routine determination on coals to be burned in stokers so that further data on the relation between the index and the degree of satisfactory performance may be obtained.

DISCUSSION

MR. RALPH E. BREWER[1] (*presented in written form*[2]).—The need for a simple, easily duplicated standard test for measuring varying degrees of "free swelling" of coals when burned in a fuel bed has long been recognized. The authors are to be commended in their attempt to accomplish this objective.

Figure 1 of the paper shows the differences to be expected in size and profile outline of coke buttons made from seven coals of widely varying rank in three different crucible tests. These differences have been proved to depend on the type of heating employed. Because of better control of heating conditions, the A.S.T.M. standard electric-furnace method for determining volatile matter should give a better distinction between coke buttons from different coals than the alternative Meker-burner method. The larger-scale British Standard Method for the Crucible Swelling Test for Coal gives larger coke buttons, and the method of reporting results enables a greater number of classifications of the coke buttons to be made. The immediate interest is in whether this latter method has any distinct advantages over the A.S.T.M. electric-furnace method in evaluating the "free-swelling" properties of coals when burned in a fuel bed.

The somewhat sketchy presentation of the test results makes it difficult to evaluate them completely. For example, the data obtained on the large number of coals listed in Tables VI, VII, and VIII of the paper leave to the conjecture of the reader the question of whether some of the coals, and which ones, are identical samples. More information in each table, with a common system of identifying samples, if applicable, would aid greatly in the study of the test results. The same argument applies to the 20 coals whose data are plotted in Fig. 5. Apparently all of the ordinate points in Fig. 5 are based up on the same standard weight of initial coal used in the separate small stoker tests of the 20 coals.

The lack of good relation between the British standard swelling number of the coal and the weight of the coke in the fuel bed at the end of the small stoker tests with the three Cary-bed coals may have a logical explanation in some recent work by the Bureau of Mines. Tests of a Lower Banner (Cary)-bed coal showed marked differences in certain properties and composition from those of Summerlee-, Sewell-, and Bakerstown-bed coals, even though all four coals are of almost identical rank. The Lower Banner coal is characterized by much higher fluidity, as determined by the Gieseler plastometer method, and by a much higher ash content than the other three coals. This coal has a petrographic composition of 36 per cent bright, 53 per cent semisplint, and 11 per cent cannel coal. In contrast, the other three coals are predominantly bright and contain no cannel coal. Analyses of the separated cannel-coal portion from the Lower Banner coal showed even higher ash and lower volatile matter contents than were present in the entire coal itself. It seems reasonable to conclude that the Lower Banner (Cary)-bed coal, because of its high fluidity, would tend to fill the voids in the fuel bed more rapidly and more completely than would less-fluid coals of similar rank. This effect would leave the air less time to burn the fuel and thus keep the voids open. Furthermore, splint and semisplint coals, and probably high-ash, low-volatile cannel coal also, are known to burn at slow rates in underfeed fuel beds and during their caking and coking do not readily open up cracks so that the air may pass freely through the fuel bed. The high fluidity, high ash content, and unusual petrographic composition of the Lower Banner (Cary)-bed coal and high ash and low volatile contents of the cannel-coal portion would thus explain the large weight of coke in the fuel bed at the end of the small stoker test of this coal. The good correlation between British standard swelling numbers of 6, or less, and the weights of coke in the fuel bed at the end of small stoker tests would indicate that such coals should be excellent fuels in small underfeed stokers.

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[1] Associate Chemical Engineer, Central Experiment Station, Bureau of Mines, Pittsburgh, Pa.

[2] Presented by permission of the Director, Bureau of Mines, U. S. Department of the Interior.

Coke buttons from the A.S.T.M. standard (electric-furnace) volatile-matter determination have been used since at least 1926 in evaluating the coking properties of various types of coals. Rose[3] has stated that "the resulting coke button will indicate whether a coal is noncoking, feebly coking, or strongly coking, but almost every condition in this test is at variance with large-scale carbonizing practice." Gradings of the buttons in terms of general coking characteristics when supplemented by the "swelling index," determined on the coke button when carbonized at 600 C. under prescribed conditions as developed by the Canadian Bureau of Mines,[4] have been applied to a large number of coals. The "agglomerating index" was proposed for indicating the dividing line between noncaking coals and those having weakly caking properties. All coals analyzed by the Bureau of Mines since November 15, 1934, have been classified according to their agglomerating properties. The agglomerating index was adopted by the A.S.T.M. in 1938 as a requisite physical property to differentiate semianthracite from low-volatile bituminous coal and also high-volatile C bituminous coal from subbituminous A coal. These few examples of the very extensive and successful application of thousands of coke-button gradings of coke buttons from the standard volatile matter determination in classifying coals according to their caking and coking properties have fully justified this practice as a desirable standard.

Mott and Wheeler[5] in England have found a relationship between the British standard crucible swelling number and the carbon and hydrogen contents of the coal. They suggested that, if certain deductions made from this relationship are supported by enough data, this swelling test might be used as a measure of coking power instead of the Sheffield Laboratory coking test. The Sheffield test, similar in principle to the Agde Damm dilatometer test extensively used in America, heats a compressed charge of coal under a load and at a rate approximating that used in a by-product coke oven. In contrast, the rate of heating in the British standard crucible swelling test is many times more rapid (approximately 300 C. per min. between 400 and 500 C.). Similar high rates of heating are used in other crucible swelling tests. Such a rapid rate of heating is impossible in commercial coke making. Mott and Wheeler pointed out that the practical differences between coals are very considerable and that the British standard crucible test may fail to distinguish between coals varying in carbon content from 87 to 91 per cent (Parr-unit coal basis), since all of these coals may give the highest swelling number, 9, that can be measured. These investigators suggested that "a wider range may be desirable than the 9 classes (each $\frac{1}{2}$ unit of swelling from 5 to 9) into which coking coals could be divided by the crucible swelling tests."

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[3] H. J. Rose, "Selection of Coals for the Manufacture of Coke," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol.74, pp. 600–636 (1926), Discussion, pp. 637–639; *Fuel*, Vol. 5, pp. 562–575 (1926), Vol. 6, pp. 41–46, 84–88 (1927).

[4] E. Swartzman, E. J. Burough and R. A. Strong, "A Laboratory Test on Coals for Predicting the Physical Properties of the Resultant By-Product Cokes," *Investigations of Fuels and Fuel Testing*, 1932, Canada Dept. Mines, Mines Branch, No. 737–3, pp. 36–57 (1934).

R. E. Gilmore, G. P. Connell and J. H. H. Nicolls, "Agglomerating and Agglutinating Tests for Classifying Weakly Caking Coals," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Coal Division, Vol. 108, pp. 255–265 (1934), Discussion, pp. 265–266.

[5] F. A. Mott and R. V. Wheeler, "The Quality of Coke," pp. 360–368, Chapman & Hall, Ltd., London (1939).

In summary, all crucible swelling tests that use very rapid rates of heating appear to be limited to broad indications of the caking and coking properties of coal, and, in their present form, such tests can only supplement but cannot replace methods using rates of heating more in keeping with plant-scale practice. In combustion processes, where the rate of heating is much higher than in gas and coke plants, crucible swelling tests appear to have merit in evaluating coals for certain types of firing. Such tests also may prove of value for the evaluation of coals to be used in horizontal and gas retorts which employ faster rates of heating than in by-product coke ovens.

MR. O. W. REES[6] (*presented in written form*).—The authors of this paper are to be congratulated for a rather thorough study of the factors influencing the crucible swelling test, duplicability of results to be obtained, and usefulness of the test. To me the results obtained by electrical heating are somewhat disappointing since this kind of heating is more easily controlled than gas burner heating. However the duplicability possible, when using gas heating, in the same laboratory and between different laboratories is quite gratifying. In our laboratory we have recently substituted a blast-type burner with grid top for the Fisher Meker type burner. Flow meters in both the air and gas lines to the blast-type burner have enabled us to duplicate conditions somewhat more readily than with the Meker-type burner with flow meter in the gas line only.

The test has been used in the Illinois Geological Survey laboratory for two or three years for the purpose of predicting the coking tendencies of coals when used in domestic underfeed stokers. Studies of eight different coals fired under one set of conditions, that is, rate of feed, air flow, etc., indicated that those coals with swelling index numbers up to about 5 gave very little coke, while those with swelling index numbers of 5 and above gave appreciable quantities of coke. The test has been of particular value in our studies of the effect of concentration or deconcentration of banded ingredients in Illinois stoker fuels on their behavior in underfeed stokers. Predictions on the basis of the test have been so successful that its use is being continued in extended studies of this kind.

MESSRS. W. A. SELVIG[7] and W. H. ODE[8] (*presented in written form*).—In our experiments at the Central Experiment Station of the Bureau of Mines with the British Standard Method for the Crucible Swelling Test for Coal we have experienced some difficulty in meeting the specified temperature inside the crucible. A Teclu burner was not available, but burners of this general type gave a maximum crucible temperature of only 755 C. when burning natural gas at a pressure of 7½ in. of water.

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[6] Chemist, Illinois State Geological Survey, Urbana, Ill.

[7] Supervising Chemist, Coal Constitution and Miscellaneous Analysis Section, Central Experiment Station, Bureau of Mines, Pittsburgh, Pa.

[8] Assistant Inorganic Chemist, Coal Constitution and Miscellaneous Analysis Section, Central Experiment Station, Bureau of Mines, Pittsburgh, Pa.

A variety of Meker-type burners were tested. Of these, only one burner gave the required crucible temperature with the gas pressure available—a Meker-type burner with a grid diameter of 1 in., so placed that the top of the grid was ⅜ in. below the bottom of the crucible. Fortunately the crucible temperature does not appear to be critical. With higher gas pressure there should be no difficulty in getting the specified temperature.

Another difficulty experienced with some coals was the lack of definite correlation between the coke buttons and the standard profiles as given in the British standard. To illustrate, if reference is made to button D, Fig. 1, of the paper, it will be seen that the profile of this button does not correspond to any of the standard profiles shown in Fig. 4. In the standard profile series, all coals which give buttons that do not swell sufficiently to touch the lid of the crucible are assigned swelling numbers of 6 or less. Button D did not touch the crucible lid, but its cross-sectional area is certainly greater than that of standard profile 6. The use of some other method of evaluating the coke buttons, such as volume measurement or cross-sectional area measurement, may make the test method more laborious, but some such modification may be necessary for troublesome coals. We have used a scheme whereby the areas of the standard profiles were plotted against the profile numbers and a curve drawn. When coke buttons of unusual shape were obtained, we drew their outlines on cross-section paper to get their areas. These areas were then referred to the curve and the nearest profile numbers recorded.

In the study of the effect of small variations in weight of sample as reported in the paper, it probably would have been better procedure if swelling numbers had been obtained on accurately weighed samples of the same coal, the weights ranging from 0.9 to 1.1 g. and these weights all made with an analytical balance. The procedured described, making weighings to 0.1 g. on an ordinary laboratory platform balance, may well have given samples that actually did not differ much in weight.

I have one question to ask Mr. Ostborg. I am a little puzzled by the statement regarding the effect of small variations in the weight of the sample. The authors state: "To determine the accuracy required in weighing the 1-g. test sample, two series of tests were run. In the first test the coal was weighed accurately to 0.1 mg. on an analytical balance, and in the second series the sample was weighed to 0.1 g. on a laboratory three-beam platform balance. The size of the coke buttons made by these two procedures was the same."

I should think that the best procedure in testing out the effect of differences in the weight of samples would be to weigh them precisely on an analytical balance. It seems to me that it is probable that, even though one set were weighed on a laboratory three-beam platform balance to 0.1 g., the actual weighings might be 1 g.

MR. H. N. OSTBORG[9] (*author's closure*).—We wish to thank Mr. Rees for his discussion, and it is gratifying to hear that the test method has been of value in determining the effect of the banded ingredients in Illinois stoker fuels on their behavior in underfeed stokers.

Mr. Selvig has raised a question concerning the method of determining the relative coke button size which has also been asked by other investigators. It is true that some coals give coke buttons that do not conform to the standard profiles. However, with some discretion concerning height and width of the button, a swelling index can be assigned to the coals. Until further investigation has shown the true significance of small variations in button size, it is believed that the size designation is not too critical and that the standard profile method is satisfactory.

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[9] Research Engineer, Battelle Memorial Institute, Columbus, Ohio.

It may be true that the effect of the weight of the sample upon the test results was not investigated as thoroughly as it might have been. However, since the paper was written, coke buttons have been made of a coal using 0.75 and 1.25-g. samples. Even with this wide deviation in weight, only a slight difference in button size was noted, and it is believed that the conclusions drawn in the paper hold true.

Mr. Brewer questions whether the British Standard Method has any distinct advantages over the A.S.T.M. electric-furnace method in evaluating the "free-swelling" properties of coals. Coke button D of Fig. 1 is made from low-volatile Pocahontas No. 3 seam coal. It can be seen that the resulting coke button was not as swollen as would be expected by comparing the buttons from the British Standard Method. Previous tests on Pocahontas coal, using the electric-furnace method, bear out these results, giving small, dense buttons instead of the large, "swollen" buttons that were expected. Coupled with the greater latitude in button size, this would appear to give the British Standard Method a distinct advantage over the electric-furnace method for determining the "free-swelling" properties of coals.

Concerning the possible duplication of results in Tables VI, VII, and VIII, in the text of the paper it states: "Table VIII gives the swelling indexes for a number of coals of various original sizes that have been measured at Battelle during the course of this investigation *in addition* to those given in Table VI." It also states that the data of Table VII "represent only a few coals, for the most part from the Pocahontas No. 3 seam, all of slack size."

The weight of the coke left in the fuel bed as given in Fig. 5 is independent of the amount of coal used in the test as the points give the weight of +1 in. coke left in the bed after an equilibrium fuel bed had been established. The depth of the fuel bed under equilibrim conditions is governed by the ratio of coal to air and by the coking characteristics of the coal.

The explanation for the variance of the relationship between the British Standard swelling index and the weight of coke in the fuel bed for the three coals from the Carey seam appears to have considerable logic, and bears out somewhat the observations of Mr. Rees.

STATISTICAL STUDY OF THE PRECISION OF METHODS FOR ANALYSIS OF COAL AND COKE

By H. H. Lowry[1] and Charles O. Junge, Jr.[1]

Synopsis

It is pointed out that a knowledge of the probable errors of analysis, which may result from following the procedures specified in A.S.T.M. Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 – 40),[2] would be of great value in any attempt to correlate performance of coals in various uses with coal analyses. Through the cooperation of nineteen organizations 176 sets of determinations, including over 32,000 analyses, were obtained. For each of these sets, the standard error of the difference between duplicate analyses on a single sample by the same laboratory was calculated by standard statistical methods. The results obtained show that the precision of the determinations varies by fairly large factors for the different laboratories and that the precision is not simply related to the "permissible difference" listed in part *B* of section 43 of Methods D 271. On the assumption that the permissible difference between duplicates should have a value such that a third determination would not have to be made more often than 50 times per thousand samples, there were calculated "most precise laboratory" and "reasonable" permissible differences and these values are compared with the present specified values. The "reasonable" permissible difference is arbitrarily defined as one which could be met by four-fifths of the laboratories that furnished the data. In some of the cases where the "reasonable" permissible difference exceeds the specified value, it is pointed out that the analyses should be made in multiplicate rather than duplicate so that the average value reported may have a precision represented by the present permissible difference.

The A.S.T.M. Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 – 40)[2] describe methods of analysis for moisture, ash, volatile matter, sulfur, carbon, hydrogen, and nitrogen and methods for determining calorific value and fusibility of coal ash. Section 43 of these methods gives the permissible differences between two or more determinations when made by the same laboratory and when made by different laboratories. The relation of these permissible differences to the probable error, or to the standard error, of the determination is now known. It was pointed out, at the meeting of Committee D-5 on Coal and Coke on March 4, 1941, that a knowledge of these errors would be of great value in any attempt to correlate performance of coals in various uses with coal analyses. Knowledge of the standard error will also permit setting up permissible differences that can be expected to be exceeded with a fixed probability, say once in twenty determinations. Following discussion of this subject, A. C. Fieldner, Chairman of Committee D-5, appointed the following personnel of a special section on Uniformity of Analytical Data of Subcommittee I on Methods of Testing: D. H. Davis, R. E. Gilmore, B. A. Landry, O. W. Rees, W. A. Selvig (*ex officio*), and H. H. Lowry, Chairman. It is with gratitude that the authors of the present report acknowledge the benefit of discussion with members of this committee of the data presented herewith; the conclusions drawn, however, are the sole responsibility of the authors. Valuable assistance has been rendered, particularly on statistical methods, by H. G. Landau, formerly a Member of the Staff of the Coal Research Laboratory.

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[1] Director, and Member of Staff, respectively, Coal Research Laboratory, Carnegie Institute of Technology, Pittsburgh, Pa.

[2] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 5.

Cooperation of the following organizations in supplying the analytical data for this study is gratefully acknowledged:

Babcock & Wilcox Co.
Battelle Memorial Institute
Bethlehem Steel Co.
Canadian Bureau of Mines
Commercial Testing and Engineering Co.
Consolidated Edison Co. of New York
Detroit Edison Co.
Foster Wheeler Corp.
Froehling and Robertson, Inc.
Fuel Engineering Co. of New York
Illinois State Geological Survey
Pennsylvania State College
Pittsburgh Coal Co.
Rochester Gas and Electric Corp.
Toledo Edison Co.
U. S. Bureau of Mines
U. S. Steel Corp. of Delaware
University of Alberta
University of Iowa

Each organization was asked to furnish the committee the results of duplicate analyses of coals for all the qualities listed in Methods D 271 which normally were made by their laboratory. It was pointed out that the analyses for the different qualities did not have to refer to the same coals. In order to avoid bias in the selection of the data to be submitted to statistical analysis, it was asked that the following procedure be followed: "Starting with a recent—preferably the latest—analysis for any given quality, report in inverse chronological order with no omission the individual analytical results up to 100 pairs. If, in selecting the analytical data this way, cases are found where it was necessary to make a third (or more) determination all three (or more) determinations should be given. For the purpose of the committee, it is desirable that the subdivisions listed in Part B of Section 43 of Methods D 271 each be regarded as a separate analytical procedure; for example, there would be given 100 pairs of observations each for moisture values below 5 per cent and for moisture values above 5 per cent. If 100 pairs of observations are not available, a smaller number can be used with a decrease in the accuracy with which the error may be determined. Also, if any change in procedure was introduced in the sequence of the 100 pairs of observations this point should be indicated. If the method used departs in any way from the A.S.T.M. standard, this should be stated, though for the purpose of this committee the deviation in procedure need not be described."

In reporting the data requested, it was asked that the following details should also be observed. "Data may be on either the as-received or air-dried basis, but the basis used should be stated. Analytical data should be given to the second decimal place, calorific values to the nearest Btu., and ash softening temperature to the nearest degree Fahrenheit. The A.S.T.M. gives three procedures for determination of sulfur, and it is, therefore, important that the method used be designated. Likewise, if the modified procedure for determination of volatile matter, Section 14(*b*), is used for only an occasional sample the data should be omitted, but if this procedure has been used on more than 30 samples the data should be given separately from the 100 pairs of observations by the standard procedure. The permissible difference of 54 F. in Part B of Section 43 of D 271 applies only to ash-softening temperature; it would be highly desirable to have 100 pairs of observations for each of the temperatures characterizing initial deformation, softening point, and fluid point, if these data are available. In connection with ash determinations, it may be assumed that there were "no carbonates present" unless the procedure used was that given in the specification for ash containing carbonates."

TABLE I.—MOISTURE.[a]

Remarks	Laboratory[b]	Min.	Max.	Σn	m	s	$\Sigma n'$	m'	s'	B'	Y'
A. < 5 PER CENT: PERMISSIBLE DIFFERENCE = 0.2 PER CENT.											
	A........	0.09	4.12	203	100	0.038	200	99	0.024	0.5	<10
	B........	0.51	2.02	96	48	0.029	96	48	0.029	0.8	<10
	C′.......	0.06	4.86	201	100	0.041	199	99	0.036	0.8	<10
	D........	0.17	4.89	202	100	0.057	194	97	0.043	0.5	<10
	E........	0.04	4.80	201	100	0.054	198	99	0.048	0	<10
	F........	0.30	4.29	200	100	0.053	200	100	0.053	0.3	<10
	G........	0.25	4.13	110	55	0.060	108	54	0.054	0.2	<10
	H′.......	0.40	3.22	200	100	0.060	200	100	0.060	0.3	<10
−8 mesh	I′.......	1.84	4.70	500	100	0.078	460	92	0.061	1.1	(<10)
	J........	0.34	4.78	200	100	0.063	200	100	0.063	0.2	<10
	K........	0.68	2.35	200	100	0.078	200	100	0.078	1.0	<20
	L′.......	2.40	4.87	200	100	0.088	200	100	0.088	0.1	<50
	M.......	0.81	4.92	200	100	0.098	198	99	0.090	0.3	<50
	N........	0.11	5.00	129	64	0.109	127	63	0.093	0.5	<50
−60 mesh	I′.......	0.6	3.3	201	100	0.103	198	99	0.096	1.2	(<50)
	O........	0.5	5.0	380	190	0.116	380	190	0.116	0.9	<100
B. >5 PER CENT: PERMISSIBLE DIFFERENCE = 0.3 PER CENT.											
	C′.......	6.28	23.85	200	100	0.049	194	97	0.039	0.7	<10
	E........	5.10	31.90	206	100	0.080	198	98	0.061	0.9	<10
	F........	4.99	23.01	200	100	0.070	200	100	0.070	0.1	<10
	J........	4.99	17.06	200	100	0.097	200	100	0.097	0.6	<10
	M.......	5.02	13.46	200	100	0.116	200	100	0.116	0.4	<10
	N........	5.13	39.80	32	16	0.165	32	16	0.165	0.1	<100

[a] The significance of the symbols is as follows:
Min. = minimum value for a single determination,
Max. = maximum value for a single determination,
Σn = total number of analyses reported,
m = number of samples,
s = standard error of difference between duplicate analyses,
$\Sigma n'$, m', and s' = the values of Σn, m, and s after eliminating samples for which the analyses showed evidence of gross error (see text),
B' = an approximate measure of the normality of distribution of errors after elimination of gross errors (see text),
Y' = calculated frequency of exceeding the A.S.T.M. "permissible" difference between duplicate determinations per thousand,
<10 = 0 to 9.9, <20 = 10 to 19.9, etc.

[b] The letters designating the cooperating laboratories are primed if it was stated that the procedure used departed in any way from A.S.T.M. Methods D 271. If a letter appears more than twice in a table, the laboratory so designated furnished more than one set of analyses. To conceal the identity of the laboratories, the assignment of letters is different in each table.

The results of this study are summarized in Tables I to VII. In addition to reporting the numerical values derived from the statistical analysis of the data by the methods described in following paragraphs, each table contains columns showing (1) the laboratory designated by a code letter which reported the data, (2) the minimum and (3) the maximum value of the quality measured, (4) the total number of analyses, and (5) the number of samples on which the analyses were made. The data are listed in each table in order of decreasing precision and the code letters were assigned, beginning with the letter "A," in the same order. If a code letter appears more than once in a table, more than one set of data were furnished by the laboratory designated by that letter. This procedure results in a different code for each table and was followed to insure that the relative precision of the collaborating organizations would not be disclosed by this report. If any laboratory indicated that the method used was not strictly the A.S.T.M. method, but "slightly modified," the code letter for that case is primed. Since some laboratories did not make specific statements that the A.S.T.M. methods were used without modification, it is possible that more of the code letters should be primed than are now so shown in the tables.

TABLE II.—ASH: PERMISSIBLE DIFFERENCE = 0.2 PER CENT.[a]

Remarks	Laboratory	Min.	Max.	Σn	m	s	$\Sigma n'$	m'	s'	B'	Y'
	A........	0.82	17.74	200	100	0.034	196	98	0.029	0.3	<10
	B........	3.24	6.02	200	100	0.048	198	99	0.045	0.8	<10
Stove coke	C........	7.78	11.92	200	100	0.071	194	97	0.049	1.5	(<10)
	D........	2.05	19.01	200	100	0.080	196	98	0.050	0.3	<10
	E........	2.05	16.37	100	50	0.052	100	50	0.052	0.1	<10
Gas coal	C........	5.32	7.41	202	101	0.063	200	100	0.059	1.0	<10
	F........	4.73	11.33	200	100	0.075	194	97	0.063	0.3	<10
Clean coal	G′.......	4.20	9.95	212	100	0.112	204	98	0.063	0.2	<10
	B........	6.25	11.92	200	100	0.072	196	98	0.064	0.2	<10
	H........	3.11	18.96	208	100	0.119	199	97	0.065	1.0	<10
Raw coal	G′.......	3.65	10.05	208	100	0.106	198	97	0.068	1.0	<10
	I.........	2.15	19.05	600	300	0.082	600	300	0.082	0.1	<20
Steam coal	C........	6.44	13.58	200	100	0.126	194	97	0.082	0.7	<20
Wet basis	J........	2.01	11.57	207	100	0.099	198	97	0.083	0.6	<20
	K′.......	1.12	28.17	233	100	0.096	225	98	0.088	1.0	<50
Raw coal	G′.......	10.05	24.45	206	100	0.118	194	97	0.090	0.1	<50
	L........	6.12	16.34	200	100	0.097	200	100	0.097	0.4	<50
	B........	11.95	60.53	200	100	0.117	198	99	0.110	0.9	<100
	M........	9.66	15.11	207	100	0.118	204	99	0.112	0.3	<100
	N........	5.06	11.93	247	100	0.159	232	96	0.133	1.6	(<200)
	O........	3.14	58.18	198	99	0.151	194	97	0.135	1.1	(<200)
	P........	3.59	23.92	118	59	0.138	118	59	0.138	0.5	<200
	Q........	5.1	10.2	204	102	0.161	204	102	0.161	0	<500
	R........	1.71	35.42	200	100	0.168	200	100	0.168	1.1	(<500)
	N........	11.89	93.41	236	100	0.218	...	...	...	...	...
Refuse and middlings	G′.......	35.20	82.25	222	100	0.477	...	...	...	...	...

[a] See footnotes to Table I.

The statistical study included first a calculation of the standard error of the difference between pairs of determinations and the results are reported in the columns of the tables headed by s. Each set of data was then examined for "gross errors," which were arbitrarily taken as those cases where the difference between duplicates exceeded 3.3 times the standard error of the difference, since if, as assumed, the errors follow the normal law of error, such values should occur with a frequency of only one in a thousand. The mathematical procedures used will be described in detail and illustrated in following paragraphs.

Following the examination of the data for gross errors and removing them from consideration if present, new values of the total number of analyses, $\Sigma n'$, the number of samples analyzed, m', and the standard error of the difference, s', were calculated and these values are also tabulated. The number of observations excluded by this procedure is readily obtained from the difference of Σn and $\Sigma n'$. From the ratio of s' to the permissible difference shown in Methods D 271, and use of probability integral tables, the frequency of occurrence of errors exceeding the permissible difference may be determined. This frequency is designated as Y' in the tables and the units are number per thousand observations. The calculated frequency, Y', is reported as falling in a given range such as <10, <20, <50, etc., where <20 signifies a frequency of 10 to 19.9, <50 signifies a frequency of 20 to 49.9, etc. This method of reporting the results of the calculated frequency of errors exceeding the permissible difference for duplicate analyses was chosen to avoid the possibility of attributing

TABLE III.[a]

Remarks	Laboratory	Min.	Max.	Σn	m	s	$\Sigma n'$	m'	s'	B'	Y'
A. BITUMINOUS COALS, STANDARD PROCEDURE: PERMISSIBLE DIFFERENCE = 0.5 PER CENT.											
	A′........	15.33	43.23	197	91	0.169	197	.91	0.169	0.6	<10
	B........	16.25	44.20	153	75	0.180	153	75	0.180	0.4	<10
	C........	15.49	43.94	155	74	0.233	149	72	0.188	0.2	<10
	D........	16.34	45.36	78	39	0.205	78	39	0.205	0.1	<20
	E........	16.80	43.78	198	99	0.206	198	99	0.206	0.1	<20
	F........	21.32	42.47	205	100	0.837	190	95	0.206	0.5	<20
	G′........	31.50	39.93	200	100	0.214	200	100	0.214	0.6	<20
	H........	18.56	47.58	192	96	0.224	192	96	0.224	1.0	<50
	I........	15.4	27.7	204	102	0.225	204	102	0.225	0.4	<50
	J′........	21.05	42.73	233	99	0.226	233	99	0.226	0.5	<50
Gas coal	K........	32.14	35.66	202	101	0.256	200	100	0.230	0.7	<50
	L........	30.8	44.8	207	100	0.273	201	98	0.249	0.4	<50
Steam coal	K........	27.16	38.31	200	100	0.252	200	100	0.252	0.6	<50
	M........	16.11	41.39	100	50	0.272	100	50	0.272	0.4	<100
	N........	15.33	38.35	110	51	0.419	102	49	0.328	0.1	<200
"Semi-anthracite"[b]	O........	7.57	19.95	113	45	0.419	109	44	0.331	0.2	<200
	P........	15.7	38.0	294	147	0.378	294	147	0.378	2.2	(<200)
	O........	14.76	37.27	230	100	0.436	230	100	0.436	0.2	<500
Wet basis	Q........	16.78	44.94	247	100	0.613	...	...	...	...	...
B. BITUMINOUS COALS, MODIFIED PROCEDURE: PERMISSIBLE DIFFERENCE = 0.5 PER CENT.											
	A′........	29.26	40.85	213	100	0.165	211	99	0.156	0.6	<10
	H........	17.85	53.44	198	99	0.188	198	99	0.188	0.5	<10
	B........	36.95	57.60	205	100	0.213	205	100	0.213	0.3	<20
	O........	25.05	43.41	220	100	0.563	216	99	0.502	0.5	<500
C. ANTHRACITE COALS AND COKES: PERMISSIBLE DIFFERENCE = 0.2 PER CENT.											
	A′........	9.86	14.24	18	9	0.066	18	9	0.066	0.5	<10
Stove coke	K........	0.36	1.71	200	100	0.090	196	98	0.080	0.4	<20
Anthracite	C........	2.64	5.89	57	26	0.103	57	26	0.103	0	<100
Coke	O........	0.42	9.51	206	100	0.131	203	99	0.121	0	<100
	B........	5.90	11.85	50	25	0.159	50	25	0.159	0.9	<500
	D........	3.35	9.65	42	21	0.215	42	21	0.215	0.3	<500
Anthracite	O........	3.10	8.75	134	55	0.518	122	52	0.265	0.5	<500
Anthracite	P........	3.6	9.8	102	51	0.320	102	51	0.320	0.6	>500
	N........	2.47	15.00	44	22	0.321	44	22	0.321	0.2	>500
Coke	L′........	7.3	23.5	107	45	0.499	92	42	0.341	0.6	>500

[a] See footnotes to Table I.

[b] These analyses were arbitrarily withdrawn from a set of analyses reported for "anthracites," see Table III*C*.

TABLE IV.—CALORIFIC VALUE, BTU. PER POUND: PERMISSIBLE DIFFERENCE = 0.3 PER CENT.[a]

Remarks	Laboratory	Min.	Max.	Σn	m	s, per cent	$\Sigma n'$	m'	s', per cent	B'	Y'
	A′	11 588	15 133	205	100	0.112	199	98	0.089	1.2	(<10)
	B	9 983	14 949	200	100	0.098	200	100	0.098	0.5	<10
	C	11 671	15 263	98	49	0.099	98	49	0.099	0.2	<10
	D	8 898	15 263	203	100	0.162	198	99	0.142	0.1	<50
	E	11 684	15 074	200	100	0.177	198	99	0.162	0.2	<100
	F	11 957	14 228	200	100	0.170	200	100	0.170	0.7	<100
	G	13 619	14 998	204	102	0.175	204	102	0.175	1.3	(<100)
	H	10 145	14 929	205	100	0.186	202	99	0.175	0.6	<100
	I	11 312	15 139	206	100	0.213	206	100	0.213	0.2	<200
Steam coal	J	13 100	14 634	200	100	0.217	200	100	0.217	0.1	<200
	K′	11 723	14 655	200	100	0.221	200	100	0.221	0.8	<200
	L	12 668	14 715	80	39	0.226	80	39	0.226	0.2	<200
	M	10 615	15 158	162	78	0.326	155	77	0.231	0.3	<200
	N	9 392	15 086	120	60	0.289	118	59	0.251	0.5	<500
	L′	12 579	14 403	202	100	0.274	202	100	0.274	0.7	<500
Wet basis	O	10 264	14 949	232	100	0.353	228	99	0.301	0.5	<500
	P	11 192	12 427	211	100	0.440	205	98	0.330	0.3	<500
	Q	7 900	14 902	258	100	0.542	258	100	0.542	0.5	>500
	R	10 735	15 325	406	182	0.863	397	179	0.736	2.2	(>500)

[a] See footnotes to Table I.

TABLE V.—ASH FUSION CHARACTERISTICS.[a]

Laboratory	Min.	Max.	Σn	m	s	$\Sigma n'$	m'	s'	B'	Y'
A. Initial Deformation Temperature in degrees Fahrenheit: Assumed Permissible Difference = 54 F.										
C′	1 971	2 832	116	58	29.0	94	47	0	∞	(<10)
A	1 847	2 568	200	100	11.2	198	99	10.0	2.1	(<10)
H	1 855	2 713	168	83	14.7	168	83	14.7	0.1	<10
E_1	2 160	2 600	200	100	15.3	200	100	15.3	0.6	<10
G	1 852	2 276	200	100	19.1	196	98	16.9	0.7	<10
F	1 760	2 770	210	100	26.9	208	99	22.8	0.9	<20
D	1 913	2 730	200	100	31.3	190	95	23.2	1.3	(<50)
E_2	2 150	2 620	100	50	28.0	98	49	24.4	0.3	<50
J	1 877	2 876	204	102	32.5	204	102	32.5	1.5	(<100)
B. Softening Temperature in degrees Fahrenheit: Permissible Difference = 54 F.										
A	1 923	2 681	200	100	10.0	200	100	10.0	1.7	(<10)
B	2 062	2 819	100	50	10.5	100	50	10.5	0.9	<10
C′	2 120	2 844	110	55	13.0	108	54	11.0	2.0	(<10)
D	1 998	2 835	200	100	14.0	194	97	12.2	0.1	<10
E_1	2 200	2 670	200	100	15.2	200	100	15.2	0.3	<10
F	1 870	2 850	210	100	22.2	206	98	17.8	0.6	<10
G	1 966	2 736	200	100	18.7	200	100	18.7	0.3	<10
H	1 900	2 800	164	81	24.4	161	80	18.9	0.3	<10
I	1 980	2 810	206	101	30.3	197	98	22.1	0.7	<20
E_2	2 200	2 670	100	50	26.6	100	50	26.6	0	<50
J	2 153	2 878	202	101	29.4	202	101	29.4	0	<100
C. Fluid Temperature in degrees Fahrenheit: Assumed Permissible Difference = 54 F.										
C′	2 384	2 832	92	46	17.0	76	38	0	∞	(<10)
A	1 982	2 860	200	100	13.8	198	99	13.1	1.4	(<10)
E_1	2 220	2 690	200	100	15.3	200	100	15.3	0.1	<10
H	1 926	2 876	162	80	26.0	159	79	17.5	0.9	<10
G	1 895	2 832	200	100	28.2	192	96	21.4	0.1	<20
J	2 525	2 900	172	86	24.6	172	86	24.6	0.3	<50
F	1 935	2 850	194	92	31.5	188	90	26.1	0.2	<50
E_2	2 220	2 690	100	50	26.8	100	50	26.8	0	<50
D	2 102	2 909	200	100	37.0	194	97	30.3	0.3	<100

[a] See footnotes to Table I.

to the actual numerical value of the calculated frequency an absolute significance.

Calculation of the frequency, Y', was made with the assumption that the errors of analysis follow the normal law of error. That this assumption is generally justified in the case of errors of measurement is supported by a considerable literature. However, the validity of the assumption was questioned by several of the collaborating organizations when the first report of the results of the statistical study of their data was sent them. Absolute justification of this assumption in each case would require an inordinate amount of computation. However, as described below, an approximate test for normality was made which yielded the values, B', shown in the tables. Unless the value of B' exceeds a value of unity, the data themselves do not give an indication of departure from normality. However, this test of normality is not rigorous and may not show all abnormal distributions. An examination of the tables indicates that values of B' greater than unity occur very infrequently. In this connection, a value of B' equals 2.1 is given

TABLE VI.—SULFUR.[a]

Remarks	Laboratory	Min.	Max.	Σn	m	s	$\Sigma n'$	m'	s'	B'	Y'
	A. ESCHKA METHOD, SULFUR < 2 PER CENT: PERMISSIBLE DIFFERENCE = 0.05 PER CENT.										
	A	0.24	1.73	201	100	0.023	198	99	0.015	1.8	(<10)
	B	0.10	0.93	206	100	0.026	198	97	0.019	1.3	(<10)
	C	0.47	1.98	106	53	0.022	106	53	0.022	0.3	<50
	D	0.11	1.99	152	74	0.023	152	74	0.023	1.2	(<50)
	E′	0.58	1.87	198	87	0.025	195	86	0.023	0.1	<50
Steam coal	F′	0.85	2.01	78	39	0.041	76	38	0.027	0.4	<100
Gas coal	F′	0.80	1.31	200	100	0.030	196	98	0.027	0.2	<100
	G′	0.37	2.02	200	100	0.030	198	99	0.029	0.4	<100
	H	1.11	1.99	132	66	0.033	132	66	0.033	0.9	<200
	I	0.07	1.84	82	40	0.042	82	40	0.042	0.8	<500
	B. ESCHKA METHOD, SULFUR > 2 PER CENT: PERMISSIBLE DIFFERENCE = 0.10 PER CENT.										
	C	2.05	9.25	94	47	0.037	94	47	0.037	1.0	<10
	A	1.99	6.87	207	100	0.061	197	97	0.042	0.4	<20
Steam coal	F′	2.03	4.30	122	61	0.052	120	60	0.045	0.7	<50
	G′	2.02	15.29	200	100	0.065	192	96	0.046	0.2	<50
	E′	2.19	5.30	26	13	0.049	26	13	0.049	0.8	<50
	D	2.11	16.86	56	26	0.049	56	26	0.049	0.6	<50
	H	2.10	3.62	66	33	0.074	64	32	0.054	0.3	<100
	I	2.01	22.74	130	60	0.086	124	57	0.054	0.7	<100
	J	2.00	14.12	200	100	0.103	192	96	0.084	0.7	<500
	C. BOMB METHOD, SULFUR < 2 PER CENT: PERMISSIBLE DIFFERENCE = 0.05 PER CENT.										
	D	0.18	1.98	179	87	0.029	176	86	0.026	0.7	<100
	K	0.57	2.01	160	80	0.030	160	80	0.030	1.3	(<100)
	L	0.50	1.56	96	48	0.037	96	48	0.037	0.9	<200
	A	0.37	2.01	204	100	0.060	195	97	0.048	0.3	<500
	M	0.43	1.70	58	29	0.061	56	28	0.051	0.6	<500
	N	0.52	1.96	160	80	0.061	158	79	0.053	0.3	<500
	D. BOMB METHOD, SULFUR > 2 PER CENT: PERMISSIBLE DIFFERENCE = 0.10 PER CENT.										
	K	2.01	3.14	44	22	0.043	44	22	0.043	0.1	<20
	D	2.08	3.38	26	13	0.053	26	13	0.053	0.2	<100
	N	2.05	4.02	40	20	0.096	38	19	0.071	0.3	<200
	A	2.06	7.79	214	100	0.118	211	99	0.108	0.3	<500

[a] See footnotes to Table I.

in Table VII for the results of determination of nitrogen by laboratory C. Generally the precision of this laboratory was excellent. However, as pointed out in our first report to the laboratory, the precision of the determination of nitrogen was of an unusually low order. The explanation offered is of general interest in connection with all of the data submitted by the different laboratories and is directly quoted herewith. "I am sorry that our nitrogen results caused your committee worry in preparing their report, particularly as there is an explanation that should have been given at the time the results were submitted. Part of the nitrogen values included were obtained by a new assistant who had considerable difficulty with this test. Since you had asked for the last 100 duplicate determinations, it was necessary to include these erratic values. As a general rule we have no particular difficulty with this determination. The error in the above case was personal and, once spotted, this operator had no further trouble in getting his determinations to check within the tolerance allowed by A.S.T.M. Methods D 271." From this explanation, it may be that certain sets of data which give values of B' greater than unity do not follow the normal law because the observations were made by more than one analyst. If each of the analysts followed the stated procedure with quite a different average error, the resulting composite data would not yield errors which were distributed according to the normal law. Furthermore, a change in equipment or in precision with time over the period during which the data

TABLE VII.—ULTIMATE ANALYSIS.[a]

Laboratory	Min.	Max.	Σn	m	s	$\Sigma n'$	m'	s'	B'	Y'
A. CARBON: PERMISSIBLE DIFFERENCE = 0.3 PER CENT.										
A	53.06	84.41	201	100	0.116	201	100	0.116	0.2	<10
B	59.40	90.01	202	100	0.138	202	100	0.138	0.1	<50
C′	54.41	88.78	206	100	0.148	203	99	0.140	0.2	<50
D′	57.46	88.23	210	100	0.296	204	98	0.214	0.2	<200
E	62.89	86.21	268	99	0.238	268	99	0.238	2.5	(<500)
F	74.09	95.20	120	60	0.447	118	59	0.268	0.3	<500
G	36.91	76.85	100	50	0.272	100	50	0.272	0.4	<500
H′	34.10	91.10	124	50	0.420	118	48	0.317	0.4	<500
I	34.32	92.72	270	100	0.505	252	95	0.409	0.9	<500
B. HYDROGEN: PERMISSIBLE DIFFERENCE = 0.07 PER CENT.										
A	2.32	6.02	201	100	0.033	201	100	0.033	0.5	<50
B	0.76	8.30	202	100	0.048	199	99	0.039	0.5	<100
C′	3.52	5.67	206	100	0.043	203	99	0.040	0.1	<100
D′	2.74	5.54	211	100	0.065	204	97	0.055	0.3	<500
I	0.59	7.10	279	100	0.117	268	97	0.080	1.4	(<500)
H′	0.34	5.78	127	50	0.143	121	48	0.088	0.8	<500
E	1.62	5.55	273	100	0.139	261	96	0.098	2.4	(<500)
F	1.44	6.04	120	60	0.136	120	60	0.136	1.8	(>500)
C. NITROGEN: PERMISSIBLE DIFFERENCE = 0.05 PER CENT.										
A	0.61	2.24	202	100	0.021	196	98	0.015	1.0	<10
B	0.58	4.58	200	100	0.025	200	100	0.025	0.3	<50
I	0.20	2.89	206	100	0.045	201	98	0.033	1.2	(<200)
D′	0.64	1.68	197	90	0.037	197	90	0.037	0	<200
C	0.75	1.76	244	100	0.099	220	95	0.060	2.1	(<500)
F	0.79	1.75	26	13	0.091	26	13	0.091	0.7	>500

[a] See footnotes to Table I.

were accumulated would also cause a distribution of errors which was not normal and yield a value of B' greater than unity.

MATHEMATICAL PROCEDURES

The determination of the standard difference was made by the procedure described by Deming and Birge.[3]

If we have several sets of measurements of quantity such that the average value differs in the different sets but the standard deviation in each set is the same, that is, *the sets of measurements have been made with the same precision*, then it is possible to combine all the values to obtain an accurate estimate of the standard deviation.

If $x_{11}, x_{12} \cdots, x_{1n_1}$ are the n_1 values in the first set

$x_{21}, x_{22} \cdots, x_{2n_2}$ the n_2 values in the second set

. . . .

$x_{m1}, x_{m2}, \cdots, x_{mn_m}$ the n_m values in the last (m th) set,

where $n_1, n_2, \cdots, n_m$ are not necessarily equal, we calculate for each set the sum of square of the deviations from its mean:

$$Q_i = \sum_{j=1}^{n_i} (x_{ij} - \bar{x}_i)^2 \quad \ldots\ldots\ldots (1)$$

($\bar{x}_i$ is the mean of the i th set).

Then our estimate of the standard deviation is:

$$\sigma^2 = \frac{Q_1 + Q_2 + \cdots + Q_m}{n_1 + n_2 + \cdots + n_m - m} \quad \ldots (2)$$

which is the sum of the squares of all the deviations, calculated from the mean of each set, divided by the total number of observations minus the number of sets.

If the sets consist of pairs, the formula and the calculations become simpler. We have:

$$n_1 = n_2 = \cdots n_m = 2$$

so that

$$n_1 + n_2 + \cdots + n_m - m = 2m - m = m.$$

Also,

$$\bar{x}_i = \frac{x_{i1} + x_{i2}}{2}$$

$$Q_i = (x_{i1} - \bar{x}_i)^2 + (x_{i2} - \bar{x}_i)^2$$

$$= \frac{(x_{i1} - x_{i2})^2}{2} = \frac{\Delta_i^2}{2} \quad \ldots (3)$$

where Δ_i is the algebraic difference between the two values in the i th pair. Then

$$\sigma^2 = \frac{\sum \Delta_i^2}{2m} \quad \ldots\ldots\ldots\ldots (4)$$

that is, the sum of the squares of the differences between the values in each pair divided by twice the number of pairs.

Illustration of the application of this procedure is relatively simple in the case of duplicate determinations. For this purpose certain data from the analysis of coal containing more than 2 per cent of sulfur will be used. First the difference between the original per cent dry sulfur determination and the duplicate is recorded for each sample.

	Original x_{i1}	Duplicate x_{i2}	Difference Δ_i	
$i = 1$	2.74	2.77	0.03	
2	2.63	2.71	0.08	
3	2.26	2.25	−0.01	
4	3.79	3.81	0.02	etc.

Next the summation of the squares of the difference is found.

$$\sum_{i=1}^{i=100} \Delta_i^2 = (0.03)^2 + (0.08)^2$$

$$+ (-0.01)^2 + (0.02)^2 + \cdots = 1.0683,$$

---

[3] W. E. Deming and R. T. Birge, "On the Statistical Theory of Errors," *Review of Modern Physics*, Vol. 6, pp. 119–161 (1934), especially pp. 162 and 158.

where

$$\Delta_i = (x_{i2} - x_{i1})$$

To find σ^2 this value is divided by twice the number of samples, m, (in this case $m = 100$ samples), or

$$\sigma^2 = \frac{\sum_{i=1}^{i=m} \Delta}{2m} = \frac{1.0683}{2 \times 100} = 0.0053415.$$

$$\therefore \quad \sigma = 0.07309$$

The value σ represents an estimate of the standard deviation of a single observation; that is, it defines the distribution of individual observations about the mean. For the purpose of this study, the difference between pairs of such observations is of greater utility than the deviations of individuals from their average. Since the difference between pairs is given by

$$\Delta = x_2 - x_1$$

its distribution is determined by the standard deviation of the x according to the theorem[4] "if $x_1, x_2, \cdots\cdot x_n$ are independent variables with variances $\sigma_1^2, \sigma_2^2, \cdots \sigma_n^2$, the function $w = c_1x_1 + c_2x_2 + \cdots\cdot c_nx_n$ has a variance $\sigma_w^2 = c_1^2\sigma_1^2 + c_2^2\sigma_2^2 + \cdots\cdot + c_n^2\sigma_n^2$. Hence, the standard deviation s of the Δ_i is given by

$$s^2 = \sigma_2^2 + \sigma_1^2 = \sigma^2 + \sigma^2 = 2\sigma^2$$

$$s = \sqrt{2}\,\sigma \qquad (5)$$

In data where more than two determinations are made on an occasional sample the standard deviation σ must first be calculated according to the unabbreviated procedure given in Eqs. 1 and 2. For those observations which were made in pairs, the differences between pairs will have the distribution defined by s, Eq. 5. Where more than two determinations were made, it is necessary to determine the standard error of the difference from the mean of the observations made by the application of theorem given above.[4] Thus, if w is this difference, defined by

$$w_n = x_j - \frac{x_1 + x_2 + \cdots x_j + \cdots x_n}{n}$$

$$= \frac{(n-1)x_j - x_1 - x_2 - \cdots - x_{j-1} - x_{j+1} - \cdots - x_n}{n} \qquad (6)$$

its standard deviation, $\bar{S}_n$, is defined by:

$$\bar{S}_n^2 = \frac{(n-1)^2}{n^2}\sigma^2 + \frac{1}{n^2}\sigma^2 + \cdots \frac{1}{n^2}\sigma^2$$

$$= \frac{(n-1)^2 + (n-1)}{n^2}\sigma^2 = \frac{n(n-1)}{n^2}\sigma^2$$

$$\bar{S}_n = \sqrt{\frac{n-1}{n}}\,\sigma \qquad (7)$$

This quantity differs from σ because it is referred to the mean of a limited number, n, of observations, which is not necessarily the same as the true mean of the distribution from which σ is measured.

Assuming that a normal distribution applies, the value Y', the number of cases in 1000 exceeding a given permissible difference x_1 can be found from a table of integrals for the normal curve. Since these tables appear in more than one form, it is to be noted that the table for

$$\frac{1}{\sqrt{2\pi}} \int_0^t e^{-\frac{t^2}{2}dt}, \qquad t = \frac{x_1}{s}$$

was used in the example below, with the equation:

$$Y' = \text{number exceeding } x_1 \text{ in } 1000$$

$$= 1000\left[1 - 2\left(\frac{1}{\sqrt{2\pi}} \int_0^{\frac{x_1}{s}} e^{-\frac{t^2}{2}dt}\right)\right].$$

The factor 2 multiplying the probability integral is used because it is necessary to consider both tails of the distribution.

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[4] J. F. Kenney, "Mathematics of Statistics," Vol. II, p. 111, Theorem VII, D. Van Nostrand Co., New York, N. Y. (1941).

For instance, from the data on sulfur greater than 2 per cent given previously, $s = \sqrt{2}\sigma = 0.10336$, $x_1 = 0.10$, and $t = \frac{x_1}{s} = 0.9675$. The table for the integral of the normal error function, $\left(\frac{1}{\sqrt{2\pi}}\int_0^t e^{-\frac{t^2}{2}dt}\right)$ gives for $t = 0.96$ a value of 0.33147 and for $t = 0.97$, a value of 0.33398. Interpolation gives for 0.9675 a value of 0.33335. Substituting this value in the equation above gives

$$Y' = 1000\,[1 - 2(0.33335)] = 333.3$$

As stated previously, before the data were studied further to check the assumption of normal distribution of errors, they were treated to remove all samples where excessively great differences indicated the occurrence of a gross error. Ordinarily this is done by removing all values whose difference from the mean exceeds 3 times the standard error. In this case, the factor (3.3) was used to reduce the probability of removing normal values. The factor (3.3) so divides the normal distribution that in less than 1 in 1000 normal values will the difference from the mean exceed 3.3 times the standard deviation, and the probability that a normal value will be removed from data of 100 duplicate determinations (a typical set of data) is $p = 0.097$. For duplicate analyses all samples where

$$|\,x_1 - x_2\,| > 3.3s = 3.3\sqrt{2}\,\sigma$$

were removed. Where n determinations were made on a sample ($n > 2$), the sample was removed if

$$|\,x_{ij} - \bar{x}_i\,| > 3.3\bar{S}_n = 3.3\sqrt{\frac{n-1}{n}}\,\sigma$$

for any one of the n determinations, x_{ij}. New values of σ, s, and $\bar{S}_n$ were calculated for data where values were removed, and the above tests repeated using the new values of s and $\bar{S}_n$. This process was continued until all such gross errors were eliminated. As a result of this procedure the values of $\Sigma n'$, m', and s' reported in the tables were obtained.

The test of the data for normality of distribution of errors was only an approximation and was based on the fact that in normal distributions one half of the values falls outside the range, $\bar{x} \pm 0.6745$ times the standard deviation. In the case considered herein, one half of the n differences, $|x_{ij} - \bar{x}_i|$ should, on the average, exceed $0.6745\,\bar{S}_n$. This distribution is satisfied only for a large number of observations, so the test obviously cannot be applied to the small number of determinations, usually two and never more than six, made on a single sample. It can have meaning only if all the data in a set, which may include determinations in duplicate, in triplicate, etc., can be combined.

Now a determination in duplicate, involving two observations, provides only one estimate of the difference from the mean. Similarly, a determination in triplicate, involving three observations, provides two estimates of difference from their mean, since the third can be calculated from the other two; and, in general, n_i observations provide $n_i - 1$ independent estimates of the deviation from their mean. Hence, the total number of independent estimates of the deviation that may be obtained from m sets of n_i observations is:

$$(n_1 - 1) + (n_2 - 1) + \cdots + (n_m - 1) = \Sigma n - m$$

If N_2 of the m sets are pairs, N_3 are in triplicate, so that:

$$2N_2 + 3N_3 + \cdots rN_r = \Sigma n$$

and the number of independent observations of the deviation is given by:

$$N_2 + 2N_3 + \cdots (r-1)N_r = \Sigma n - m$$

it may be expected, for a normal distribution, that on the average:

$\frac{1}{2}(2\ N_2)$ determinations on pairs will have $|w_2|$ exceeding $0.6745\ \bar{S}_2$

$\frac{1}{2}(3\ N_3)$ determinations on threes will have $|w_3|$ exceeding $0.6745\ \bar{S}_3$

$\frac{1}{2}(4\ N_4)$ determinations on fours will have $|w_4|$ exceeding $0.6745\ \bar{S}_4$, etc.

where the w's and $\bar{S}$'s are defined by Eqs. 6 and 7. Of these, however, only $\frac{1}{2}(N_2)$, $\frac{1}{2}(2\ N_3)$, $\cdots$ will be independent observations, so that the total number of independent observations of differences from the mean greater than the appropriate $0.6745\ \bar{S}_n$ will be:

$$\frac{1}{2}[N_2 + 2N_3 + \cdots + (r-1)N_r] = \frac{1}{2}\left[\sum n - m\right]$$

In any particular case, the total number of observations lying outside the appropriate $0.6745\ \bar{S}_n$ limits will differ from this average value by an amount ϵ which, itself, has a binomial distribution about zero with standard deviation

$$\sigma_\epsilon = \frac{1}{2}\sqrt{\sum n - m}$$

In less than one out of twenty sets of normal data will $|\epsilon|$ exceed $2\sigma_\epsilon$ ($p = 0.0455$). Since there are usually far less than twenty sets of data in any group, this probability is small enough to suggest the use of $2\ \sigma_\epsilon$ as a limit for $|\epsilon|$. The value B listed in the tables is:

$$B = \frac{|\epsilon|}{2\sigma_\epsilon},$$

so if B is less than one (that is, $|\epsilon| < 2\sigma_\epsilon$), the data do not give an indication of departure from normal.

DISCUSSION OF RESULTS OF THE ANALYSIS

As stated previously, Tables I to VII summarize the results of the study of the data submitted. The significance of the symbols used as headings of the columns has been pointed out in preceding paragraphs. All values for Y', the expected frequency of differences between duplicate analyses exceeding the permissible difference of Method D 271, for sets of data which, according to the test for normality of distribution of errors, yielded a value of B' greater than unity are bracketed. It would possibly be more logical to omit the derived figures for precision of the tests for these sets of data, though the infrequency of occurrence of values of B' considerably greater than unity renders this procedure doubtful in significance.

Also, the method of treating the data from laboratories which have stated that the procedures used are those of the A.S.T.M. "slightly modified" is open for discussion.

It will be noted that the precision of the different determinations varies from laboratory to laboratory. In attempting to derive any general conclusion on the precision of the determinations, it must be recognized that the standard error of difference, s or s', also has a standard deviation which is proportional to the reciprocal of the square root of the number of observations minus the number of the samples included in the analysis. This means that the values reported for s' and Y' are determined with a lower degree of accuracy where $(\Sigma n' - m')$ is small than where it is large. In drawing any final conclusions regarding precision of the analytical procedures, the different values of the s' should probably be weighted proportionately to $\sqrt{\Sigma n' - m'}$.

In Table I *A* are reported the results on moisture determinations of coals containing less than 5 per cent moisture obtained from 15 laboratories. The standard error of difference between duplicates, s', ranges from 0.024 to 0.116 per cent and the expected frequency of differences exceeding the permissible difference ranges from <10 to <100 times per thousand. Analytical procedures are frequently controlled, when statistical methods are used, to give the equivalent of a "permissible difference" which will be exceeded not more than fifty times per thousand; this criterion will be used arbitrarily throughout the following discussion as a basis of questioning the reasonableness of the existing values of "permissible difference" given in Methods D 271. A permissible difference equal to 1.96 s' would yield a value of $Y' = 50$, so it is evident that the value specified at present is clearly on the liberal side. Only one laboratory would not be able to qualify for this precision. However, data submitted by one laboratory are excluded from the table since the precision obtained was of an entirely different order of magnitude, the values of s' and Y' being 0.309 and >500, respectively. In assessing the value of statistical studies of errors such as this, it is of interest that the laboratory which submitted these data on receiving our first report examined the procedure followed in the moisture determination and located the source of inaccuracy in the low sensitivity of the balance used for the weighings. A new set of data were then obtained with more accurate weighing and the results which were obtained were analyzed and are reported in Table I *A* under the code letter I′. The new set of data show a threefold improvement in the precision of the determination.

It is not the purpose of this report to recommend changes in the permissible differences specified by the A.S.T.M. for the analytical determinations on coal and coke. However, in each case it will be pointed out what permissible difference could be met by four-fifths of the laboratories, where the permissible difference is a value which would not be exceeded more frequently than fifty times in a thousand duplicate analyses. In this connection, where a third value is determined because the difference between duplicates exceeds 1.96 s', that is, $Y' = 50$, the average of all three values should have greater accuracy than the average of the two closest values if errors are distributed according to the normal law of error. Only if one of the three values differs from the average of all three by an amount greater than $\frac{3.3}{\sqrt{3}}\,s'$, that is 1.91 s', should it be not used in computing the final value to report. When four or more values are to be averaged, the factor times the permissible difference for determining whether one value should be discarded can be computed from consideration of statistical theory as outlined in the section of this report on mathematical procedures.

Examination of the data in Table I *A* indicates that reduction of the permissible difference from 0.2 to 0.15 per cent might be reasonable on the basis outlined in the immediately preceding paragraph.

Only six laboratories reported analyses of coals containing more than 5 per cent moisture (Table I *B*). The precision with one exception is greater than would be expected with a value of 0.3 per cent for the permissible difference. Reduction of this value to 0.2 per cent might be reasonable.

The permissible difference given in Methods D 271 for duplicate determinations of ash in coal and coke is 0.2 per cent, *unless the coal contains pyrites and carbonates and the ash over 12 per cent,* in which case the permissible difference is 0.5 per cent. In submitting the data which were used to obtain the results shown in Table II, no laboratory indicated that the samples analyzed contained carbonates and that there was justification, therefore, for use of the higher permissible difference. However, several laboratories, on receipt of the first report on the statistical study of their data, have stated that it was a fair assumption that in their coals, which contained more than 12 per cent ash, carbonates were present. This is unquestionably the case for laboratory R, Table II, which is located in a region where the coals frequently contain appreciable amounts of carbonate. However, since the data were not segregated into those containing carbonate and those not containing it, the data were treated uniformly with those from other laboratories.

The data of Table II would seem to support a proposal that the permissible difference for ash determinations should increase with the ash content; this is particularly well illustrated by consideration of the data submitted by laboratories B and G. It seems doubtful that the requirements of Methods D 271 should be applicable to data on samples containing ash in amounts as high as 82.25 per cent reported by laboratory G on one set of samples or as high as 93.41 per cent reported by laboratory N and therefore complete analysis of these data is not reported in Table II. For the remaining sets of data the standard difference ranges from 0.029 to 0.168 per cent. However, if one also excludes from consideration the data which yielded a value of B' greater than unity and hence may include errors not distributed according to the normal law, it would appear that a reduction in the permissible difference to 0.15 per cent for coals and cokes containing less than 10 per cent ash would cause little hardship; for higher ash coals this value might be raised to 0.25 per cent and the restriction on proved presence of carbonates removed.

It appears from Table III *A* that most laboratories have little difficulty in determining volatile matter by the standard procedure without exceeding the permissible difference of 0.5 per cent in more than fifty times per thousand. When the modified procedure is used, Table III *B*, only one of the four laboratories is out of line. However, the conditions are quite different for the precision of the determination of volatile matter in anthracites and cokes. Part of this difficulty may be attributed to inclusion of data on volatile matter for low temperature cokes which were of little or no commercial importance when the permissible differences were established. In this latter category, Table III *C*, only two of the ten laboratories report results which would accord with the established permissible difference of 0.2 per cent. Several of the laboratories indicated difficulty with determinations of volatile matter content of anthracites and low temperature cokes. The data are inadequate to permit a justification of using the same permissible difference for anthracites and low-temperature cokes as for "coke," which undoubtedly meant high-temperature coke when the specification was established. It may be provisionally concluded that a permissible difference of 0.2 per cent is a reasonable value for coke, that the value should be about 0.5 per cent for anthracite, and about 0.7 per cent for low-temperature coke. These conclusions are particularly uncertain because of the relatively small number of analyses reported—most of which were segregated from the original reports on the basis of volatile matter but with confirmation from the reporting laboratory.

It has frequently been stated that the calorific value of a coal is one of the most precise determinations that can be made on it. The data submitted, as shown in Table IV, do not bear out this statement. In only four of the nineteen sets of data would the existing permissible difference not require at least a third determination to meet the requirements of Methods D 271 more than fifty times in a thousand. From the data submitted, a reasonable value of permissible difference would be 0.5 per cent. Even the higher limit suggested would require frequent redeterminations of calorific value for four of the laboratories if the specifications were strictly followed. In view of the importance of the calorific value in determining the relative use values of coals, the data submitted would indicate the need for frequent determination in multiplicate rather than in duplicate so that a high precision of the mean of the values obtained on a given sample will be assured. There also exists the possibility that a thorough examination of the calorimetric procedures now being followed by laboratories D to R inclusive might increase their precision to values comparable to that obtained by laboratories A to C.

The data reported on ash fusion characteristics are summarized in Table V. Although Methods D 271 contains a permissible difference only for the ash-softening temperature (*ST*), Table V *B*, each laboratory was asked for data on determinations of both the initial deformation (*IDT*) and fluid temperatures (*FT*) as stated in the introduction. Examination of the data reported indicated that in many cases duplicate determinations could not be strictly so considered since the frequency of exact check determinations was inordinately high. Determination of ash-fusion temperatures is, to a much greater degree than other determinations, dependent on subjective factors. Accordingly, each laboratory was asked to state whether duplicate determinations as reported were made *in the same furnace at the same time*. This was certainly the case for all of the data of laboratory C and some of the data for laboratories B and E_1, and the data submitted by these laboratories should be given little weight in drawing conclusions on the precision of the tests. Laboratory E submitted two sets of data designated as E_1 and E_2. As a general rule, this laboratory does not run ash-fusion determinations in duplicate and the first set of data were obtained by the operator generally running two samples of the same ash in the same furnace together. In the second set of data, two separate determinations were made, and the operator was not informed of which samples were duplicate. There is a marked difference in the calculated precision for the two sets of data, though it is not as large as might have been anticipated. If the data for laboratories B, C, and E_1 are, therefore, not considered true duplicates, only the data of laboratories E_2, F, G, and H for *IDT*, of laboratories D, E, F, G, H, I, and J for *ST*, and of laboratories D, E_2, F, G, H, and J for *FT* may be regarded as significant for the purposes of this report, since the other data are excluded by failure to meet the test for normality of distribution of errors. Within these limitations, the standard differences observed range from 14.7 to 24.4 F. for *IDT*, 12.2 to 29.4 F. for *ST*, and 17.5 to 30.3 F. for *FT*. There does not appear any marked difference in the precision of the determination of the several characteristic temperatures. The permissible difference of 54 F. appears to be a reasonable value for the ash-softening temperature, and also for the other characteristic fusion temperatures.

In Table VI there are given the data submitted for determination of the precision of analysis of coals and cokes for sulfur by the Eschka and bomb methods. For each of these methods, the data were separated into samples containing less than 2 per cent sulfur and those containing more than 2 per cent sulfur. In general, it appears that the Eschka method is the more precise. The data would appear to indicate, on the previously stated basis, that a reasonable permissible difference for determination of sulfur by the Eschka method in coals containing less than 2 per cent sulfur would be 0.06 per cent, while that for coals containing more than 2 per cent sulfur would remain unchanged at 0.10 per cent. However, for the bomb method the values should probably be about 0.10 per cent and 0.15 per cent, respectively.

From the data shown in Table VII, it would appear that, with one exception, or possibly two, none of the laboratories reporting data on ultimate analysis can carry through the specified procedures for the determinations and meet the permissible difference 95 per cent of the time. It may be suggested that the methods of D 271 for determination of carbon, hydrogen, and nitrogen are not closely enough specified. The data submitted would indicate that the permissible difference for carbon might be raised to about 0.5 per cent, that for hydrogen to about 0.15 per cent, and that for nitrogen to about 0.10 per cent. An alternative, as in the case of calorific value, would be to accept the necessity of making multiplicate rather than duplicate determinations so as to obtain the desired precision of the reported average. It may be that duplicate analyses obtained in combustion procedures could be obtained with greater precision if they were made more regularly. Analyses made after the combustion train has stood idle for a time are apt to be erratic until the system becomes regularized.

SUMMARY AND CONCLUSIONS

A statistical study has been made of data submitted by nineteen laboratories on the analytical qualities of coal and coke for which methods of determination and permissible differences between duplicate determinations are given in A.S.T.M. Methods D 271 – 40.[2] If one is willing to accept the thesis that a "reasonable" permissible difference between duplicate analyses is one which would not require a third determination by more than four-fifths of the laboratories more than fifty times in a thousand, then the values shown below should be used rather than the present values. For purposes of comparison, there is also shown below the permissible difference that could be met by the laboratory making the particular determination with the greatest precision.

	"Most Precise Laboratory" Permissible Difference, per cent	"Reasonable" Permissible Difference, per cent	Specified Permissible Difference, per cent
Moisture			
<5 per cent	0.05	0.15	0.2
>5 per cent	0.08	0.2	0.3
Ash			
No carbonates	....	....	0.2
Carbonates	....	....	0.5
<10 per cent	0.06	0.15	
>10 per cent	0.10	0.25	
Volatile Matter			
Bituminous coals, etc.			
Standard procedure	0.35	0.5	0.5
Modified procedure	0.30	0.5	0.5
Anthracites and coke	....	....	0.2
Anthracites	0.2	0.5	0.2
Coke (high temperature)	0.15	0.2	0.2
Coke (low temperature)	0.7	0.7	0.2
Calorific Value	0.2	0.5	0.3
Ash Fusion Characteristics			
Initial deformation temperature	30 F.	54 F.	....
Softening temperature	25 F.	54 F.	54 F.
Fluid temperature	35 F.	54 F.	....
Sulfur			
Eschka method			
<2 per cent	0.04	0.06	0.05
>2 per cent	0.07	0.10	0.10
Bomb method			
<2 per cent	0.05	0.10	0.05
>2 per cent	0.08	0.15	0.10
Ultimate Analysis			
Carbon	0.25	0.5	0.3
Hydrogen	0.07	0.15	0.07
Nitrogen	0.03	0.10	0.05

DISCUSSION

Mr. O. W. Rees[1] (*presented in written form*).—This paper deserves the careful study of all those interested in coal analysis. For the purpose of gaining a knowledge of the errors to be expected in reported coal analytical data as related to performance of coals in various uses, the study is quite useful. In addition, information of value to the coal analyst has been gained in regard to the precision to be expected when using A.S.T.M. standard procedures.

It is interesting to note the variation in precision of results secured by various laboratories when using standard procedures for which rigid tolerances are provided. These variations may indicate that the provisions of the method are not specific enough or that present specifications are not being followed as closely as could be. In any case this study should prompt each laboratory to check closely on the details of procedure now used as compared to the standard A.S.T.M. procedures.

Strictly speaking, a statistical study of the precision to be expected when using standard procedures, assuming a normal distribution, should exclude those data not showing a normal distribution. However, as stated by the authors, the relatively few cases where the data submitted depart from a normal distribution would seem to make such exclusion of doubtful significance. In addition it would seem that data obtained by modified procedures should not be included in a study of the precision of standard procedures. A study of Tables I to VII will show that the precisions of modified procedures show no distinct differences from those of standard procedures. However, it should be borne in mind that while a modified procedure may give satisfactorily checking duplicates, these results may vary from results obtained by rigidly following standard procedures.

In referring to Table II, which presents data on ash determination, the authors suggest that the data indicate the "proposal that the permissible difference for ash determinations should increase with ash content." May this not vary with the composition of the ash rather than with the amount? In support of this suggestion a case of a coal containing considerable amounts of calcium and magnesium carbonates but practically no pyritic sulfur came to my attention. When ashed no difficulty was experienced in obtaining satisfactory results, although the ash value was rather high. On the other hand certain coals containing calcite and pyrite in appreciable quantities have given trouble although the ash content was not as high as in the previously mentioned sample.

Mr. W. A. Selvig[2] (*presented in written form*).—The paper represents a lot of hard work in assembling and evaluating coal analysis data submitted by many cooperating laboratories and the results of the statistical study should prove of considerable value to Committee D-5 on Coal and Coke.

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[1] Chemist and Head, Analytical Division, Illinois State Geological Survey, Urbana, Ill.

[2] Supervising Chemist, Coal Constitution and Miscellaneous Analysis Section, Central Experiment Station, U. S. Bureau of Mines, Pittsburgh, Pa.

It is quite possible that the present permissible difference of 0.2 per cent between duplicate determinations of volatile matter in anthracite is too stringent and should be increased. Likewise, it seems advisable to have different tolerances for high- and low-temperature cokes.

One of the rather surprising results of the study is the relatively poor showing made in the calorific value determination. This is a very important determination in evaluating coal. It is questionable whether the permissible difference between duplicate determinations should be raised as high as 0.5 per cent as this would mean 70 Btu. for a 14,000-Btu. coal. The present tolerance of 0.3 per cent should not be difficult to meet if the attention is paid to the calorimetric procedure that its importance warrants.

I believe it reasonable to allow a larger permissible difference in duplicate determinations for the bomb-washing method for sulfur than for the Eshka method. Inherently the bomb-washing method may be quite precise, but it is not as "fool-proof" as is the old reliable Eshka method. In routine work, sufficient care is often not used in washing out the bomb completely. Years ago when we first investigated the bomb-washing method the results submitted by various cooperating laboratories in determining sulfur on the same coals were generally so unsatisfactory that it was believed that the bomb-washing method could not be recommended as a standard method. Later tests gave satisfactory check results when the cooperating laboratories were cautioned concerning the importance of following strictly the specified procedure. This is mentioned because certain methods are generally more reliable when employed for routine work because they are not so much affected by variations in laboratory technique and the urge to "cut corners" by speeding up the procedures.

Possibly the good showing made in the ash fusion test is because the cooperating laboratories realize that this is a comparatively difficult test and requires strict adherence to the specified procedure as published in the standard methods.

It is not surprising that difficulties were experienced in the ultimate analysis in determining carbon and hydrogen, as this analysis requires a higher degree of manipulative skill and good condition of the combustion train, both of which are difficult to attain if determinations are made at irregular intervals. Here possibly it would be better not to raise the permissible differences between duplicate determinations but to make multiplicate rather than duplicate determinations to obtain the required precision.

MR. O. O. MALLEIS[3] (*by letter*).—This study of actual differences between duplicates in determinations in the laboratory analysis of coal and coke should be of considerable value not only in the evaluation of the precision of the various methods but also in pointing out possible shortcomings in procedure, apparatus, or technique. Comments in this discussion will be confined to two determinations, namely, volatile matter and calorific value.

In the case of the determination of volatile matter in anthracite and high- and low-temperature cokes, it is generally known that too fine grinding of the sample will be the cause of erratic results. Rose[4] pointed out the effect of fineness in case of the determination of volatile matter in high-temperature coke. It is the experience of the writer that the analysis portions, for determination of volatile matter in anthracite and high- and low-temperature cokes should have as much "near 60-mesh" material as possible to obtain in regular preparation. In case of several low-temperature cokes, it seemed necessary to increase the coarseness in order to have as much "near 8-mesh" material as possible. Lower values and smaller differences between duplicates were obtained in the latter case.

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[3] Director, Inspection and Technical Service, Bituminous Coal Producers Board for District No. 8, Cincinnati, Ohio.

[4] Harold J. Rose, "Some Conditions Affecting the Accuracy of the Determination of Volatile Matter in Coal and Coke," *American Gas Association Monthly*, Vol. 3, p. 625 (1921).

Possibly this study, by Lowry and Junge, would indicate the desirability of reinvestigating the entire procedure to see if some modification, such as the coarseness of the analysis portion, may be made so that the present permissible differences would not have to be materially altered in case of the determination of volatile matter in anthracite and high- and low-temperature coke.

In case of calorific value, the study of Lowry and Junge might indicate that the permissible differences should be increased. This, however, I believe, would be unfortunate as it is the writer's experience that many of the calorific values of coal used in the trade are unsatisfactory and that there is a need for improvement of the quality of calorific values in many instances. The writer's own experience, in a laboratory making intermittent determinations, was that there was little difficulty checking within the permissible difference. Undoubtedly, if this study only served as an aid toward improving the general quality of the important determination of calorific value, this study will have served a most useful purpose.

A RAPID METHOD OF ESTIMATING THE A.S.T.M. RING-AND-BALL SOFTENING POINT OF ASPHALTS

By A. B. Hersberger[1] and C. Overbeck[1]

Synopsis

The method presented has been used in our laboratories for the past six months. The A.S.T.M. softening point of asphalt is estimated in approximately 10 min. starting with a liquid sample. The range of 90 to 250 F. is covered. Variations in results between operators and laboratories are of the same order as those obtained with the standard test.

The method is based on a relationship between the A.S.T.M. softening point and the time required for the asphalt, molded in a standard ring loaded with ball, to flow 1 in. when immersed in the proper test bath. This relationship was established from data obtained on both straight-run and oxidized asphalts.

The A.S.T.M. softening point[2] is one of the most commonly determined properties of asphalts. This softening point is not a melting point in the sense of representing a change in state, but is a temperature, determined by means of an arbitrary and rigidly defined method, representing some phase in the gradual change in asphalt consistency as the temperature is raised. The test is used as a control both in the manufacture and processing of asphalt and in laboratory work. Frequently the relatively long time (approximately 1½ hr.) required to make a determination is a great disadvantage.

The test described here enables the A.S.T.M. softening point of asphalts in the range of 90 to 250 F. to be estimated in approximately 10 min., starting with a liquid sample and some knowledge of the temperature range in which the softening point may lie. The test is based on a relationship between the time required for the asphalt, molded in a standard A.S.T.M. ring loaded with ball, to flow 1 in. when immersed in the proper test bath maintained at constant temperature. Three test bath temperatures are required to cover the range of softening points between 90 and 250 F. The procedure for preparing and conditioning the test specimens before immersing in the bath is rigidly defined. Very little special equipment is required, and the cooling temperatures necessary to condition the test specimens are those available in most asphalt testing laboratories.

Description of the Test

Apparatus:

The essential test apparatus consists of the following:

1. (*a*) Test baths maintained at 130

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[1] Senior Chemist, and Junior Engineer, respectively, Research and Development Dept., The Atlantic Refining Co., Philadelphia, Pa.

[2] Standard Method of Test for Softening Point of Bituminous Materials (Ring and Ball Method) (D 36 – 26). 1939 Book of A.S.T.M. Standards, Part II, p. 417.

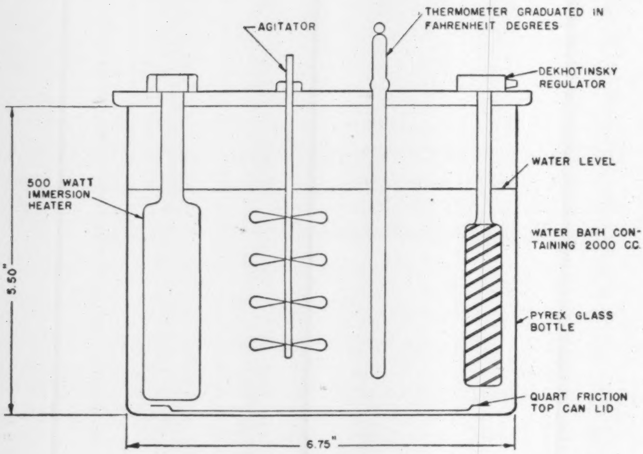
AGITATOR
THERMOMETER GRADUATED IN FAHRENHEIT DEGREES
DEKHOTINSKY REGULATOR
WATER LEVEL
500 WATT IMMERSION HEATER
WATER BATH CONTAINING 2000 CC.
5.50"
PYREX GLASS BOTTLE
QUART FRICTION TOP CAN LID
6.75"

Fig. 1.—Test Bath.

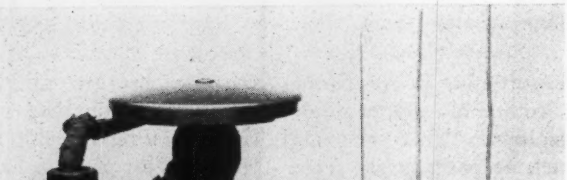

Fig. 2.—Assembled Apparatus.

± 0.5 F., 180 ± 0.5 F., and 230 ± 1 F. A satisfactory design is shown in Fig. 1. Water is used for the 130 and 180 F. temperatures while glycerine is used for the 230 F. temperature.

(*b*) Water baths maintained at 115 ± 0.18 F., 77 ± 0.18 F., and 37 ± 1.0 F. The 115 and 77 F. baths are normal A.S.T.M. penetration test baths.[3] The 37 F. temperature can be conveniently maintained by using a 600-ml. beaker filled with 3 in. of water and set in a bed of cracked ice.

2. Normal softening point rings and balls as specified in A.S.T.M. softening point test (D 36 – 26).[2]

3. An amalgamated Tobin bronze block 3 by $1\frac{1}{8}$ by $1\frac{1}{8}$ in. drilled on one side to hold two softening point rings. The holes are $\frac{3}{32}$ in. in diameter and their centers are $\frac{7}{8}$ in. from the ends and $\frac{5}{16}$ in. from one side of the block.

The assembled apparatus is shown in Fig. 2.

4. Timer or stop watch to read in 0.2 sec.

TABLE I.—TEST PROCEDURE.

	For Asphalts in the Range of 90 F. to 140 F. Softening Point.	For Asphalts in the Range of 135 F. to 185 F. Softening Point.	For Asphalts in the Range of 180 F. to 250 F. Softening Point.
1	Place the softening point rings on the amalgamated Tobin bronze block and pour the liquid asphalt sample into two softening point rings in a manner to leave an excess on cooling.		
	Pouring temp., 200 to 350 F.	Pouring temp., 250 to 450 F.	Pouring temp., 350 to 450 F.
2	Allow molded samples to remain in air at room temperature exactly 1 min.		
3	Place block and molded samples in 77 F. bath exactly 4 min.	Place block and molded samples in 77 F. bath exactly 4 min.	Place block and molded samples in 115 F. bath exactly 4 min.
4	Remove the block and molded sample from the bath and shave the excess asphalt from the rings with a slightly heated spatula.		
5	Immediately immerse the molds in the 37 F. bath and allow to stand exactly 3 min.	Immediately immerse the molds in the 37 F. bath and allow to stand exactly 3 min.	Immediately immerse the block and molded samples in the 115 F. bath and allow to stand exactly 3 min.
6	Remove the molds from the bath and immediately place the steel balls on the surface of the sample as near to the center of the sample as possible (steel balls at room temperature).		
7	Immediately immerse the molds in the 130 F. bath and start the timer at the same instant.	Immediately immerse the molds in the 180 F. bath and start the timer at the same instant.	Immediately immerse the molds in the 230 F. bath and start the timer at the same instant.
8	Record the times required for the bituminous material to reach the bottom of the bath.		
9	Read estimated A.S.T.M. softening point from the conversion tables.		

Test Procedure:

The test procedure is summarized in Table I.

Precautions:

1. The successful use of the test is dependent on the exactness of the time the sample is allowed to remain at the temperatures noted.

2. Gentle stirring, just sufficient to maintain uniform temperature throughout the test baths, should be used, as excessive agitation causes turbulence which affects the accuracy of the determination.

3. In holding the rings containing the molded asphalt in the test baths, the surface of the asphalt should be horizontal.

4. The timer should be started the instant the molds are immersed in the test bath.

Accuracy:

Duplicate determinations should be made and the time in seconds obtained should not differ by more than 7 per cent based on an average value.

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[3] Standard Method of Test for Penetration of Bituminous Materials (D 5 - 25), 1939 Book of A.S.T.M. Standards, Part II, p. 412; Part III, p. 185.

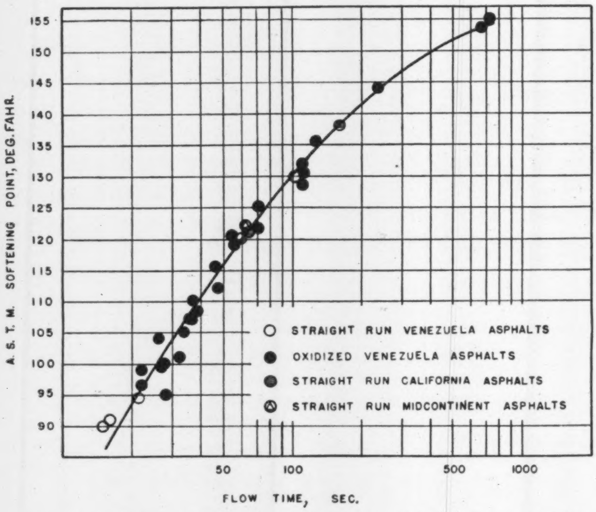

FIG. 3.—A.S.T.M. Softening Point *versus* Flow Time in Rapid Test. Test bath temperature, 130 F

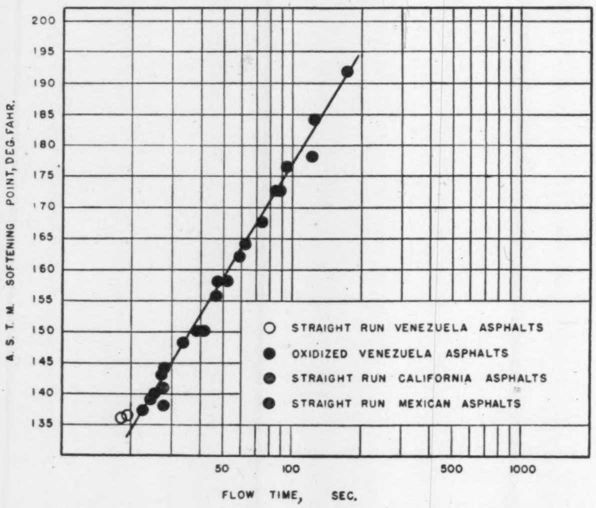

FIG. 4.—A.S.T.M. Softening Point *versus* Flow Time in Rapid Test. Test bath temperature, 180 F.

DISCUSSION

The use of three different test bath temperatures was found necessary since at low temperatures the high softening point asphalts require excessively long times (30 min.) to flow the specified distance of 1 in., which defeats the purpose of the test. With test bath temperatures high enough to cause rapid flow of high softening point asphalts, the flow times for the soft materials are too short to be readily measured with sufficient accuracy. The three test bath temperatures finally selected enable all determinations within the softening point range of 90 to 250 F. to be made in approximately 10 min. The intermediate softening point range covered by the 180 F. bath overlaps the other two ranges by five degrees, that is, asphalts having softening points between 135 and 140 F. and 180 and 185 F. may be determined satisfactorily in two of the three baths. The procedure for conditioning the test specimens before immersing in the test baths has been established so that uniform results are obtained.

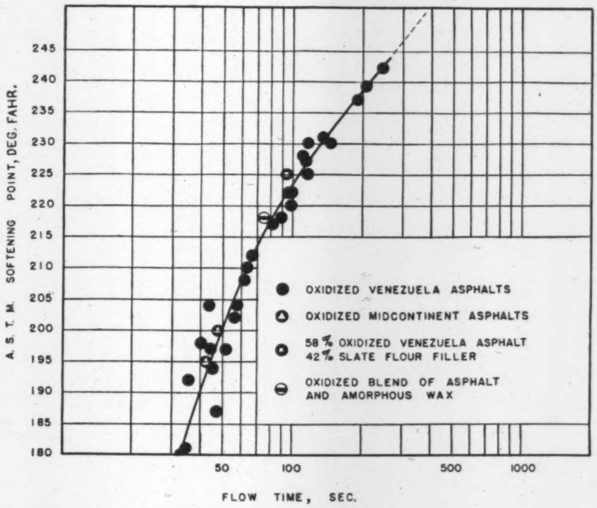

Fig. 5.—A.S.T.M. Softening Point *versus* Flow Time in Rapid Test. Test bath temperature, 230 F

The data from which the conversion tables are constructed are shown in Figs. 3 to 5. The diameter of the plotted points represents a variation of ±0.9 F. in the A.S.T.M. softening point. Both the A.S.T.M. softening points and the seconds of flow obtained in the rapid test are an average of two determinations. The test specimens for both the A.S.T.M. and rapid tests were poured at the same time from one container to avoid any difference in the softening points of the test specimens due to "history of the sample."

The asphalts tested were, for the most part, prepared from Venezuela crude oil; however, a number of asphalts from other sources were included, as well as one mixture of asphalt and slate flour.

By examining the figures mentioned above it is seen that the majority of the determinations lie on smooth curves. The reason for some of the points not lying on the curve is undoubtedly due to an error in one or both of the determinations. Check results were not made on those samples deviating from the average curves inasmuch as it would have required remelting small samples of asphalt which might have altered their softening points. However, it is believed the errors are as likely to be in the A.S.T.M. determinations as in the rapid tests.

Neither the crude oil source nor the method of preparation has any bearing on the relationship between the two tests. The conversion figures appear in Table V.

The percentage differences between

determinations are shown in Table II for a total of 85 determinations. Of this total, 65 or 76.5 per cent fall within a maximum deviation of not more than 7 per cent which is equivalent to approximately ±0.9 F. in the A.S.T.M. softening point over the entire range.

This test has been in use in two laboratories of this company for a number of months with excellent success. Variations between operators in the different laboratories are shown in Table III. The A.S.T.M. softening points of two other asphalts determined by the five different operators are shown in Table IV. All of these figures are based on two determinations. Examination of these data shows that the results obtained by the rapid test can be accepted with the same confidence as actual A.S.T.M. determinations.

TABLE II.—VARIATIONS IN EIGHTY-FIVE DUPLICATE DETERMINATIONS MADE WITH THE RAPID TEST.

Percentage of Total Tests	Difference Between Times of Flow for Two Determinations, per cent
18.8	less than 1
31.8	1 to 4
25.9	4 to 7
9.4	7 to 10
14.1	over 10

TABLE III.—RESULTS OBTAINED BY TWO LABORATORIES USING THE RAPID SOFTENING POINT TEST.

Sample	A.S.T.M. Softening Point deg. Fahr. (Obtained by Laboratory 1)	A.S.T.M. Softening Point Estimated by the Rapid Test, deg. Fahr.				
		Laboratory 1	Laboratory 2			
			Operator 1	Operator 2	Operator 3	Operator 4
A....	151	150	148	...	147	150
B....	159.5	159	161	160	159	161.5

TABLE IV.—A.S.T.M. SOFTENING POINTS REPORTED BY DIFFERENT OPERATORS.

Sample	A.S.T.M. Softening Point, deg. Fahr.				
	Operator 1	Operato. 2	Operator 3	Operator 4	Operator 5
C......	150	152	151	150	149
D......	127	130	129	127	127

TABLE V.—CONVERSION TABLE.

Seconds at 130 F. to A.S.T.M. Softening Point		Seconds at 180 F. to A.S.T.M. Softening Point		Seconds at 230 F. to A.S.T.M. Softening Point	
Reading, sec.	Softening Point, deg. Fahr.	Reading, sec.	Softening Point, deg. Fahr.	Reading, sec.	Softening Point, deg. Fahr.
18	90	21	135	33	180
18.5	91	21.5	136	34	181
19	92	22.5	137	34.5	182
20	93	23.5	138	35	183
21	94	24	139	35.5	184
22	95	25	140	36	185
22.5	96	26	141	37	186
23	97	27	142	37.5	187
24	98	28	143	38	188
25	99	29	144	39	189
26	100	30	145	39.5	190
27	101	31.5	146	40.5	191
28	102	32.5	147	41	192
29	103	34	148	42	193
30	104	35.5	149	43	194
31.5	105	36.5	150	44	195
33	106	38	151	45	196
34.5	107	39.5	152	46	197
36	108	41	153	47	198
37	109	42.5	154	48	199
39	110	44	155	48.5	200
40.5	111	46	156	50	201
42	112	47.5	157	51	202
44	113	49	158	52	203
46	114	51	159	54	204
48.5	115	53	160	55	205
50.5	116	55	161	56	206
53	117	57	162	58	207
55	118	59.5	163	60	208
58	119	62	164	61	209
61	120	63	165	63	210
64	121	66	166	65	211
67	122	69.5	167	66	212
70	123	72	168	69	213
74	124	75	169	71	214
77	125	78	170	73	215
81	126	80.5	171	76	216
85	127	83	172	78	217
90	128	86.5	173	82	218
95	129	90	174	84	219
100	130	93.5	175	87	220
105	131	97	176	91	221
110	132	101	177	95	222
118	133	105	178	98	223
125	134	108.5	179	102	224
130	135	112	180	107	225
140	136	116.5	181	111	226
150	137	121	182	117	227
159	138	125.5	183	122	228
168	139	130	184	128	229
178	140	133	185	134	230
				140	231
				145	232
				152	233
				160	234
				170	235
				175	236
				185	237
				195	238
				205	239
				215	240
				225	241
				240	242
				250	243
				265	244
				275	245
				290	246
				300	247
				320	248
				340	249
				350	250

DISCUSSION

MR. I. E. MANNING[1] (*presented in written form*).—As chairman of Subcommittee B-5 on Softening Point of Committee D-4 on Road and Paving Materials, I have gathered informally several criticisms of the paper from members of my subcommittee and others interested in the softening point test.

These composite criticisms, briefly and generally stated, are as follows:

1. The method appears to be attractive where a large number of softening point tests are run and results are needed quickly. This would apply to control testing.
2. The method, as presented, is not considered suitable as a replacement of, or a supplement to, the current A.S.T.M. Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (D 36 - 26),[2] which latter test is considered satisfactory for referee and ordinary testing purposes.
3. Extra testing equipment is an undesirable feature of the method presented.
4. The limited number of sources of bituminous materials used in the data indicates the possibility that conversion curves for other sources may be necessary.

MESSRS. A. B. HERSBERGER[3] and C. OVERBECK[3] (*authors' closure*).—In answer to Mr. Manning's comments, I should like to point out that this test is not intended as a replacement for the Standard Ring and Ball Softening Point Test (D 36 - 26). It is a rapid means of obtaining an estimate of the ring and ball softening point. The additional equipment required for this test is relatively small and seems to us to be entirely justified in view of what is accomplished.

It is true that before the test can be considered applicable to all materials, numerous other products must be examined, especially coal tars. However, we do feel that the test can be applied successfully to asphalts of petroleum origin.

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[1] Chief Chemist, Bayonne Refinery, Standard Oil Co. (New Jersey), Bayonne, N. J.

[2] 1939 Book of A.S.T.M. Standards, Part II, p. 417.

[3] Senior Chemist and Junior Engineer, respectively, Research and Development Dept., The Atlantic Refining Co., Philadelphia, Pa.

A STATISTICAL COMPARISON OF THE METHODS FOR THE DETERMINATION OF THE OXIDATION TENDENCY OF INSULATING OILS[*]

BY A. E. FLOWERS[1] AND S. A. FRUCHTMAN[1]

A very large amount of cooperative laboratory work has been carried out by the members of Subcommittee IV on Liquid Insulation, of Committee D-9 on Electrical Insulating Materials, during the past 16 yr. In each laboratory tests were run on duplicate samples of oils, by a number of different methods, with an attempt to follow the same set of directions in each laboratory. There has been little, if any, interchange of personnel, or of apparatus.

There have been considerable variations in the reported values, both within a given laboratory in repeat or check tests, and much larger variations between laboratories.

It became difficult to evaluate these results, because of the large amount of data, and of the usual, but considerable, variations therein, common to these highly sensitive chemical reactions. A purely statistical analysis of all the data has therefore been prepared, and is presented herewith, covering the period 1926 to 1942 with the following objects in mind:

1. To obtain the deviation from the grand average of the results of the laboratories cooperating in A.S.T.M. oxidation tests of oils, for each series of tests.
2. To obtain the deviation from the average of the check results run within a given laboratory, for each series of tests.
3. To compare the reproducibility of the different methods of test.
4. To compare the reproducibility of a given method of test in different years of test.
5. To determine the effect of the duration of test and the amount of sludge formed, on the reproducibility of the Sludge Accumulation Tests.

The methods of test for oxidation characteristics of insulating oil included in this study, arranged in their chronological order, are:

1. The Snyder life test.
2. The Sligh oxidation test.
3. The sludge accumulation test without catalyst.
4. The sludge accumulation test with a copper catalyst.
5. The high-pressure oxygen bomb accumulation test.

Detailed descriptions of the test methods themselves have been adequately given elsewhere and are not repeated in this report due to lack of space. The principal characteristics of each test are, however, recapitulated in Table I.

Statistically, these methods of test may be arranged as follows, in the order of their reproducibility as indicated by the average coefficient of variation between laboratories:

Method	Coefficient of Variation, per cent
1. Sligh oxidation test	12
2. High-pressure oxygen bomb accumulation test	25
3. Snyder life test	25
4. Sludge accumulation test with catalyst (1939 to 1942)	35
All sludge accumulation tests with catalyst (1933 to 1942)	50
5. Sludge accumulation tests without catalyst	50

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[*] The presentation of this paper at the annual meeting was sponsored by Committee D-9 on Electrical Insulating Materials.

[1] Engineer, and Chemical Engineer, respectively, The De Laval Separator Co., Poughkeepsie, N. Y.

Comments:

1. It is readily seen that, in the sludge accumulation tests, using a catalyst to reduce the length of time has not altered the reliability of the results from the statistical standpoint of reproducibility.

2. It may also be seen upon examination of the detailed tabulations in the body of this report that there is a very much greater variation between laboratories than within a laboratory. Statistically, this may mean that the directions may not be as complete as they could be, or that the actual experimental variations in conditions are greater than the supposed limits.

3. It seems that persons conducting tests are not as well instructed or as careful as they are supposed to be, or that there is need for interchange of personnel or apparatus.

TABLE I.—PRINCIPAL CHARACTERISTICS OF THESE TEST METHODS FOR THE EVALUATION OF INSULATING OILS—A.S.T.M. NAPHTHA

Summary of Subcommittee IV Tests, for Evaluating Insulating Oils.

Name of Test	Size of Sample	Temperature of Test, deg. Cent.	Duration of Test	Air or Oxygen	Rate of Air or Oxygen Admission	Catalyst Used	Container	Criterion for Evaluating Oil	Reference
Snyder.......	500 ml.	120	See heading "Criterion"	Air	Surface exposure	None	Glass containers, 600-ml. beakers	Days required for sludge formation to occur	A.S.T.M. *Proceedings*, Vol. 25, Part I, p. 365 (1925)
Sligh..........	10 g.	200	2.5 hr.	Oxygen	Surface exposure	None	Special glass flasks	Weight of sludge precipitated with A.S.T.M. naphtha	A.S.T.M. *Proceedings*, Vol. 27, Part I, p. 461 (1927)
Sludge Accumulation with No Catalyst....	35 ml.	120	21 to 56 days	Air	1 to 2 cu. ft. per hr.	None	Test tube	Weight of sludge precipitated with A.S.T.M. naphtha	Section B Circular letter of Dec. 28, 1931
Sludge Accumulation with Copper Catalyst....	25 ml.	120	3 to 14 days	Air	0.5 liter per hr. per tube of oil	Copper	Test tube	Weight of sludge precipitated with A.S.T.M. naphtha	A.S.T.M. *Proceedings*, Vol. 41, p. 413 (1941)
High pressure Oxidation..	50 ml.	140	24 hr.	Oxygen 250 psi.	Surface exposure	None	Test tube placed in a bomb	Weight of sludge precipitated with A.S.T.M. naphtha	A.S.T.M. *Proceedings*, Vol. 41, p. 413 (1941)

4. Increasing or shortening the length of time of exposure to oxidizing conditions does not markedly change the reproducibility of sludge accumulation tests. The occurrence of greater or lesser quantities of sludge does not appreciably change the reproducibility of sludge accumulation tests. Intermediate conditions for time and quantity of sludge seem to give slightly better reproducibility.

5. Oils rated by number of days before appearance of sludge, as in the Snyder life test, show more reproducible results for those oils that required a greater number of days for sludge to appear. This seems to be largely due to the arithmetic relation of the unit of measurement (one day) to the number of days reported as the "life."

6. This analysis of the data from the records of Section B on Sludge Tests of

Subcommittee IV was carried out solely as a statistical study of the numerical data. As such it did not concern itself directly with questions of the significance of the different methods nor their suitability for classification of oils for purchase, or for service ratings.

METHOD

1. *To Obtain the Deviation from the Grand Average of the Results of the Laboratories Cooperating in A.S.T.M. Oxidation Tests of Oils:*

To obtain the deviation from the grand average, the arithmetic average of the several tests made within each laboratory was first calculated. Then, the "standard deviation" for all laboratories from the grand average, was calculated, using the *individual laboratory averages*.

The standard deviation of a set of numbers, X_1, X_2, X_3, is the square root of the average of the squares of the deviations of the numbers from their average, $\bar{X}$, that is:

$$\text{Standard deviation} = \sqrt{\frac{(X_1 - \bar{X})^2 + (X_2 - \bar{X})^2 + \cdots + (X_n - \bar{X})^2}{n}} \qquad (1)$$

Equation 2, derived from Eq. 1, is more convenient to use in computations:

$$\text{Standard deviation} = \sqrt{\frac{X_1^2 + X_2^2 + \cdots + X_n^2}{n} - (\bar{X})^2} \ldots\ldots (2)$$

Using this equation, the standard deviation is obtained by dividing the sum of the squares of the numbers by n, subtracting the square of their average, and then extracting the square root.

The coefficient of variation is obtained by dividing the standard deviation by the grand average ($\bar{X}$), or:

$$\text{Coefficient of variation} = \frac{\text{Standard deviation}}{\bar{X}} \times 100 \ldots\ldots\ldots\ldots (3)$$

2. *To Obtain the Deviation from the Average of the Check Results Run Within a Given Laboratory:*

The deviation from the average of the results run within a given laboratory is taken as the sum of the absolute deviations of the results from their own average, $\bar{X}$, divided by the number of determinations, n. This value is the percentage mean deviation and is:

$$\text{Percentage mean deviation} = \frac{|X_1 - \bar{X}| + |X_2 - \bar{X}| + \cdots + |X_n - \bar{X}|}{n\bar{X}} \times 100$$

The percentage mean deviation is obtained for the results of each laboratory, and individual laboratories may be so compared with other laboratories. To compare the deviations of the results from the individual laboratory average, for the several methods, the percentage mean deviation for each laboratory is averaged for each series of tests.

By the use of the above methods, the coefficient of variation of the results of the cooperating laboratories from the grand average, and the average percentage mean deviation of the check results were found for each oil tested. However, more than one oil was tested in most of the cooperative tests, and for the purpose of better comparsion of the several methods, the coefficient of variation and percentage mean deviation were averaged for all oils tested by a given method.

REPRODUCIBILITY OF RECENT SLUDGE-ACCUMULATION TESTS USING COPPER CATALYST (1939–1942)

General:

This sludge-accumulation test is essentially a measure of the sludging tendency of an oil, accelerated by a supply of treated air, a copper catalyst, and a temperature of 120 C. An oil or air

TABLE II.—SLUDGE ACCUMULATION TESTS OF 1940–1941 (14 LABORATORIES—174 DETERMINATIONS).[a]

	3-day Test					7-day Test					14-day Test				
	Sludge, per cent by weight		Coefficient of Variation Between Laboratories, per cent	Mean Deviation Within Laboratories, per cent		Sludge, per cent by weight		Coefficient of Variation Between Laboratories, per cent	Mean Deviation Within Laboratories, per cent		Sludge, per cent by weight		Coefficient of Variation Between Laboratories, per cent	Mean Deviation Within Laboratories, per cent	
	Min.–Max.	Average		Min.–Max.	Average	Min.–Max.	Average		Min.–Max.	Average	Min.–Max.	Average		Min.–Max.	Average
Oil A.....	0.022–0.060	0.039	28	0.5–10.0	4	0.062–0.150	0.109	21	1.7–11.4	6	0.083–0.329	0.191	29	0–4.4	2
Oil B.....	0.051–0.150	0.101	26	0.5–13.7	5	0.163–0.485	0.252	34	1.4–14.8	7	0.759–1.037	0.526	52	0.9–40.8	12
Average		0.07	27		5		0.18	28		7		0.36	40		7

[a] The values for "Sludge, per cent by weight" in this and subsequent tables are based on the mean value reported by laboratories. Each laboratory made two determinations and reported the mean of these two determinations. In a few laboratories a third determination was run and the mean of the three reported.

In many reports from laboratories, both or all three values were reported as well as the mean. The percentage mean deviation within laboratories is based upon the separate determinations. The maximum and minimum values reported here and in the other tables, are the highest and lowest values for any individual test value from any of the reporting laboratories.

TABLE III.—SLUDGE ACCUMULATION TESTS OF 1942 (6 LABORATORIES—64 DETERMINATIONS).

	3-day Test					7-day Test					14-day Test				
	Sludge, per cent by weight		Coefficient of Variation Between Laboratories, per cent	Mean Deviation Within Laboratories, per cent		Sludge, per cent by weight		Coefficient of Variation Between Laboratories, per cent	Mean Deviation Within Laboratories, per cent		Sludge, per cent by weight		Coefficient of Variation Between Laboratories, per cent	Mean Deviation Within Laboratories, per cent	
	Min.–Max.	Average		Min.–Max.	Average	Min.–Max.	Average		Min.–Max.	Average	Min.–Max.	Average		Min.–Max.	Average
Oil A.....	0.017–0.066	0.048	41	2.3–18.9	9	0.057–0.155	0.107	27	3.2–16.3	7	0.111–0.228	0.181	20	3.6–10.1	6
Oil B.....	0.076–0.102	0.102	25	0.8–24.5	14	0.146–0.285	0.217	23	3.6–26.9	10	0.208–0.479	0.388	26	1.8–20.9	7
Average..........		0.08	33		12		0.16	25		9		0.28	23		7

TABLE IV.—SLUDGE ACCUMULATIONS FOR 1940-1941—FOR THE 6 LABORATORIES WHO RECHECKED THEIR RESULTS IN 1942 ONLY (70 DETERMINATIONS).

	3-day Test					7-day Test					14-day Test				
	Sludge, per cent by weight		Coefficient of Variation Between Laboratories, per cent	Mean Deviation Within Laboratories, per cent		Sludge, per cent by weight		Coefficient of Variation Between Laboratories, per cent	Mean Deviation Within Laboratories, per cent		Sludge, per cent by weight		Coefficient of Variation Between Laboratories, per cent	Mean Deviation Within Laboratories, per cent	
	Min.–Max.	Average		Min.–Max.	Average	Min.–Max.	Average		Min.–Max.	Average	Min.–Max.	Average		Min.–Max.	Average
Oil A.....	0.022–0.054	0.041	27	0.5–3.4	2	0.062–0.150	0.111	26	2.1–10.2	7	0.083–0.329	0.205	45	1.1–3.9	3
Oil B.....	0.051–0.121	0.096	28	0.7–13.7	6	0.173–0.485	0.299	36	2.8–14.6	7	0.259–1.037	0.656	48	2.5–4.8	15
Average..........		0.07	28		4		0.21	31		7		0.43	47		9

bath is used for heating the oil, contained in a special test tube. After 3, 7, or 14 days of this accelerated oxidation, the sludge is precipitated by A.S.T.M. naphtha, and the oxidized oil filtered, using a Gooch crucible.

Calculated Results:

Sludge Accumulation Tests of 1940–1941.—The data for the calculated results shown in Table II are taken from the June 20, 1941 report of Section B of Subcommittee IV of Committee D-9. The results are based on the values of 14 laboratories and a sum of 174 individual determinations.

Sludge Accumulation Tests of 1942.—The data from Table III are taken from the February 14, 1942 report of Section B. These tests were run on the same oils tested in 1940–1941 and were performed by six rechecking laboratories (three of which had obtained high values in the tests of 1940–1941, one, low values, and two, average values). The results are based on the values of these six laboratories and a sum of 64 individual determinations.

Sludge Accumulation Tests of 1940–1941—for the Six Rechecking Laboratories of 1942 Only.—In order better to compare the performance of the six rechecking laboratories in 1942 with their performance in 1940–1941, there is presented in Table IV, values of percentage sludge by weight, coefficient of variation between laboratories, and percentage deviation of the check results within a laboratory for the results obtained by these laboratories in 1940–1941.

The values are for these six laboratories only, and the data are taken from the June 20, 1941 report of Section B, and are based on a sum of 70 individual determinations.

Sludge Accumulation Tests of 1940.—The data for Table V are taken from the June 21, 1940 report of Section B and are for the 1940 results of 13 laboratories, and a sum of 120 determinations.

TABLE V.—SLUDGE ACCUMULATION TESTS OF 1940 (13 LABORATORIES—120 DETERMINATIONS).

	7-day Test					14-day Test				
	Sludge, per cent by weight		Coefficient of Variation Between Laboratories, per cent	Mean Deviation Within Laboratories, per cent		Sludge, per cent by weight		Coefficient of Variation Between Laboratories, per cent	Mean Deviation Within Laboratories, per cent	
	Min.-Max.	Average		Min.-Max.	Average	Min.-Max.	Average		Min.-Max.	Average
Oil A	0.05 -0.40	0.24	41	1.4–18.3	7	0.29–0.98	0.49	45	0.2–40.3	12
Oil B	0.025–0.25	0.13	51	0.4–18.4	5	0.05–0.34	0.20	49	0.7–36.1	11
Average		0.19	46		6		0.35	47		12

TABLE VI.—SLUDGE ACCUMULATION TESTS OF 1939 (10 LABORATORIES—28 DETERMINATIONS) (ONE OIL).

7-day Test					14-day Test				
Sludge, per cent by weight		Coefficient of Variation Between Laboratories, per cent	Mean Deviation Within Laboratories, per cent		Sludge, per cent by weight		Coefficient of Variation Between Laboratories, per cent	Mean Deviation Within Laboratories per cent	
Min.-Max.	Average		Min.-Max.	Average	Min.-Max.	Average		Min.-Max.	Average
0.07–0.16	0.11	29	3.1–9.4	6	0.10–0.40	0.21	36	0.6–23.2	10

Sludge Accumulation Tests of 1939.—The data in Table VI are taken from the October 18, 1939 report of Section B, and are for the 1939 results of 10 laboratories, and a sum of 28 individual determinations.

The Effect of the Duration of Test and the Amount of Sludge Formed on the Reproducibility of Recent Sludge-Accumulation Tests Using Copper Catalyst:

Sludge Accumulation Tests of 1940–1941 (Table II).—From Table II it is seen that the coefficient of variation between laboratories does not vary greatly for the lighter sludging oil A, while it increases from 26 per cent for the 3-day test to 52 per cent for the 14-day test for the heavier sludging oil B.

Sludge Accumulation Tests of 1942—Six Recheck Laboratories (Table III).—From Table III, it is seen that the coefficient of variation between laboratories for the lighter sludging oil A decreases from 41 per cent at 3 days to 20 per cent at 14 days, while it remains rather constant for the heavier sludging oil B.

Sludge Accumulation Tests of 1940–1941—for the Six Laboratories Who Rechecked Their Results in 1941 (Table IV).—From Table IV it is seen that the coefficient of variation between laboratories for oil A increases from about 27 per cent at 3 and 7 days to 45 per cent at 14 days, while for oil B it increases from 28 per cent at 3 days to 48 per cent at 14 days.

Sludge Accumulation Tests of 1939–1940 (Table V).—Table V shows that the coefficient of variation between laboratories for the lighter sludging oil (B in this case) decreases very slightly, from 51 per cent at 7 days to 49 per cent at 14 days, while it increases slightly from 41 per cent at 7 days to 45 per cent at 14 days, for the heavier sludging oil.

Sludge Accumulation Tests of 1939.—From Table VI, it is seen that the coefficient of variation increases slightly from 29 per cent at the 7-day test to 36 per cent at the 14-day test.

Discussion.—(Values of Table IV are not included in this discussion, as they are statistically included in Table II.)

The increase of the coefficient of variation of the 1940–1941 test results for oil B is largely due to some exceptionally high results of three of the laboratories. These results were not reproduced in 1942 when rechecking their test values for percentage sludge for the 14-day test, which were half of their values of 1940–1941. In recheck tests on oil A, the two laboratories reporting highest results, obtained values only two-thirds of their 1940–1941 test values for the 14-day test. It seems as though the error involved in the measurement of the sludge becomes especially apparent when large amounts of sludge are being measured, but may be reduced by the proper precautions.

Increasing the days of test seems to be of more value for the lighter sludging oil than for the heavier sludging oil. Increased days of test produced little change in the coefficient of variation for the lighter sludging oil A, in tests of 1940–1941 (Table II), while the coefficient of variation was increased for the heavier sludging oil B. Also, while little change was produced by increased days of test, in tests of 1942 (Table III) for the heavier sludging oil B, the coefficient of variation was thereby reduced for the lighter sludging oil A.

Data of Tables V and VI for tests of 1940 and 1939 indicate little change in the coefficient of variation with increase in days of test.

The Reproducibility of the Sludge Accumulation Tests of 1940–1941 Compared with the Reproducibility of the Same Tests Run on the Same Oils in 1942 by Six Rechecking Laboratories.—It has been mentioned above that the recheck

TABLE VII.—SUMMARY OF RECENT SLUDGE ACCUMULATION TESTS USING COPPER CATALYST (1939-1942) TABLES I, II, III, IV, V—(386 DETERMINATIONS).

Test Year	Number of Laboratories	Number of Determinations	Number of Oils Tested	Days of Test	Sludge, average per cent by weight	Coefficient of Variation Between Laboratories, per cent		Average Mean Deviation Within Laboratories, per cent	
					Min.-Max.	Min.-Max.	Average	Min.-Max.	Average
1940–1941	14	174	2	3–14	0.04–0.53	21–52	32	2–12	6
1942	6	64	2	3–14	0.05–0.39	20–41	27	6–14	9
1940–1941*	6*	70*	2*	3–14*	0.04–0.66*	26–48*	35*	2–15*	7*
1940	13	120	2	7–14	0.13–0.49	41–51	46	5–12	9
1939	10	28	1	7–14	0.11–0.21	29–36	33	6–10	8
General Average	6–14	386			0.04–0.66	20–51	35	2–15	8

* In computing the values for the general average, separate values for Table III were omitted, and the values are the arithmetic averages of Tables I, II, IV and V.

tests of 1942 showed better reproducibility. From the summary of Table VII, it is seen that the over-all coefficient of variation between laboratories, for the six recheck laboratories, was reduced from 35 per cent in 1940–1941 to 27 per cent in 1942.

Comparison of the Reproducibility of the Recent Sludge Accumulation Tests.—From Table VII, it is seen that the average coefficient of variation was 33 per cent for tests of 1939, 46 per cent for tests of 1940, 32 per cent for tests of 1939–1941, and 27 per cent for tests of 1942. These results, in general, show a trend towards a decrease in the coefficient of variation between laboratories.

THE REPRODUCIBILITY OF EARLY AND MISCELLANEOUS SLUDGE ACCUMULATION TESTS USING COPPER CATALYST (1933–1939).

The reproducibility of recent sludge accumulation tests using a copper catalyst (1939–1942) has been discussed above. However, other sludge accumulation tests using a copper catalyst have been performed in previous years, but with some modifications.

Sludge Accumulation Tests Using Copper Catalyst in Which Air is Blown Through the Oil:

Results of sludge accumulation tests using a copper catalyst in which air is blown through the oil (Table VIII) have been reported in the October 18, 1939 report of Section B. In this procedure, the oil was oxidized by passing the conditioned air through the oil at 120 C. in the presence of copper. The sludge was precipitated by A.S.T.M. naphtha, and the oxidized oil filtered, using a Gooch crucible.

The reproducibility of this supplementary procedure may be compared with that of the regular method (see Table VI). The average coefficient of variation by the supplementary procedure, for the 7- and 14-day test periods, was 23 per cent, while for the same oil treated in the same year (1939) by the regular procedure, the corresponding figure was 33 per cent. Of course, it must be remembered that the standard procedure was used by 10 laboratories, and the supplementary procedure by

TABLE VIII.—SLUDGE ACCUMULATION TESTS USING A COPPER CATALYST—AIR BLOWN THROUGH THE OIL (3 LABORATORIES—6 DETERMINATIONS).

The data for the difference of the check results within any laboratory were not given.

Days of Test	Sludge, per cent by weight		Coefficient of Variation Between Laboratories, per cent
	Min.-Max.	Average	
7	0.09–0.23	0.15	39
14	0.23–0.26	0.25	6
Average		0.20	23

only 3. As might be expected, the average percentage sludge (by weight) was somewhat higher when determined using the supplementary procedure in which air is blown through the oil, the corresponding figures being 0.15 and 0.11 for the 7-day period, and 0.25 and 0.21 for the 14-day period.

Sludge Accumulation Tests Using Copper Catalyst (1933–1935):

In these tests the oil was oxidized, using a temperature of 120 C. and a supply of untreated air. After a period of 3 to 16 days, the sludge was precipitated by naphtha and determined by centrifuging. This method principally differs from that of Table VII in that the air was untreated, and the sludge was determined by centrifuging. The data from which the following results have been calculated are from the reports of July 3, 1935 and October 20, 1933 of Section B.

TABLE IX.—SLUDGE ACCUMULATION TESTS USING COPPER CATALYST (1933) (5 LABORATORIES—100 DETERMINATIONS).

The average coefficient of variation is 59 per cent. Check results were not available.

Oil	3-day Test			6-day Test			9-day Test			13-day Test			16-day Test		
	Sludge, per cent by weight		Coefficient of Variation Between Laboratories, per cent	Sludge, per cent by weight		Coefficient of Variation Between Laboratories, per cent	Sludge, per cent by weight		Coefficient of Variation Between Laboratories, per cent	Sludge, per cent by weight		Coefficient of Variation Between Laboratories, per cent	Sludge, per cent by weight		Coefficient of Variation Between Laboratories, per cent
	Min.–Max.	Average		Min.–Max.	Average		Min.–Max.	Average		Min.–Max.	Average		Min.–Max.	Average	
A-33	0.001–0.039	0.018	68	0.004–0.139	0.047	80	0.006–0.157	0.081	68	0.016–0.117	0.086	44	0.054–0.238	0.129	60
B-33	0.005–0.038	0.016	70	0.014–0.100	0.043	79	0.050–0.226	0.114	66	0.053–0.196	0.129	44	0.073–0.350	0.200	53
C-33	0.010–0.186	0.059	120	0.033–0.120	0.079	44	0.063–0.290	0.167	56	0 099–0.397	0.246	21	0.215–0.502	0.334	29
D-33	0.021–0.057	0.037	45	0.039–0.543	0.199	93	0.133–0.756	0.334	76	0.248–0.500	0.368	27	0.296–0.591	0.480	26
Average......		0.033	76		0.092	74		0.174	67		0.207	34		0.286	42

In Table IX, for tests performed in 1933, the values are only for those five laboratories who followed the procedure exactly. In Table X, for tests performed in 1934–1935, results of the Detroit Edison Laboratories have not been included because of a very wide divergence from the results of other laboratories.

The average coefficient of variation for these sludge accumulation tests using a copper catalyst run during 1933–1935, was about 60 per cent, as compared with an average coefficient of variation of 35 per cent for the tests using copper catalysts run during 1939–1942. The oils tested were in the same sludge range, and these results indicate a refinement in technique in the interval between 1935–1939. The average percentage mean deviation of the check results run within a laboratory, was 11 per cent for the tests of 1934–1935, as compared to 8 per cent for the tests of 1939–1942. Sludge accumulation tests, using no catalyst, (Tables XII to XIV) run in the same period as these tests, using a catalyst, showed better reproducibility. The average coefficient of variation for these tests was 50 per cent for the interval 21 to 56 days, and 40 per cent for test periods greater than 40 days.

TABLE X.—SLUDGE ACCUMULATION TESTS USING COPPER CATALYST (1934-1935) (7 LABORATORIES—200 DETERMINATIONS).

The average coefficient of variation is 62 per cent. Check results were given only for results of 3 laboratories.

Oil	3-day Test					6-day Test					9-day Test					13-day Test					16-day Test				
	Sludge, per cent by weight		Coefficient of Variation Between Laboratories, per cent	Mean Deviation Within Laboratories, per cent		Sludge, per cent by weight		Coefficient of Variation Between Laboratories, per cent	Mean Deviation Within Laboratories, per cent		Sludge, per cent by weight		Coefficient of Variation Between Laboratories, per cent	Mean Deviation Within Laboratories, per cent		Sludge, per cent by weight		Coefficient of Variation Between Laboratories, per cent	Mean Deviation Within Laboratories, per cent		Sludge, per cent by weight		Coefficient of Variation Between Laboratories, per cent	Mean Deviation Within Laboratories, per cent	
	Min.-Max.	Average		Min.-Max.	Average	Min.-Max.	Average		Min.-Max.	Average	Min.-Max.	Average		Min.-Max.	Average	Min.-Max.	Average		Min.-Max.	Average	Min.-Max.	Average		Min.-Max.	Average
A-34...	0–0.018	0.009	73	0–11	4	0.012–0.082	0.029	81	0–11.5	7	0.018–0.220	0.087	77	3–6	5	0.025–0.447	0.153	86	3.5–8	6	0.055–0.482	0.241	65	8–17	12
B-34...	0.001–0.038	0.018	72	11–20	17	0.009–0.200	0.064	93	0–13	7	0.037–0.455	0.198	65	6–12	9	0.052–0.645	0.327	59	7–10	8	0.077–1.035	0.509	57	8–16	11
C-34...	0.003–0.055	0.025	61	0–32	14	0.025–0.303	0.112	79	0–31	14	0.045–0.436	0.226	51	7–38	23	0.095–0.860	0.374	62	6–20	10	0.115–1.124	0.494	62	5–34	14
D-34...	0.030–0.080	0.051	33	0–16	7	0.086–0.290	0.156	40	2–10	6	0.127–0.600	0.333	45	3–26	14	0.213–1.05	0.671	42	4–12	7	0.235–1.45	0.976	43	10–12	12
Average.......		0.026	60		11		0.090	73		11		0.281	59		13		0.381	62		8		0.555	57		12

The tests of 1934–1935 (Table X) show no significant variation of the coefficient of variation related to the duration of test, while the tests of 1933 (Table IX) show a noticeable decrease in the coefficient of variation for the 13-day test. This may have no real significance.

Sludge Accumulation Tests Using Copper Catalysts (1937):

These tests were run to note the effect of the method of sludge determination upon the reproducibility. The oil was oxidized at a temperature of 120 C., using treated air. After a 7- and 14-day period of oxidation, the sludge was precipitated by A.S.T.M. naphtha and the weight determined by:

(*a*) Centrifuging and weighing.
(*b*) Use of a fritted glass crucible.
(*c*) Use of a Gooch crucible.

The percentage sludge of the oxidized oil, the reproducibility of the results between laboratories, and the average percentage mean deviation of the check results for these tests are given in Table XI. The coefficient of variation of the results between laboratories (considering the results of all laboratories) seemed to be considerably higher than for other tests using copper as a catalyst (see Tables II to X). In an effort to determine a cause for this difference, the results of the laboratories reporting the highest and lowest results were directly compared and it was found in some cases that the results submitted by one laboratory were 100 times that submitted by the other. Accordingly, further calculations were made for (1) all laboratories except the highest one, and (2) all laboratories except the highest and lowest, and the results given in Table XI were obtained.

TABLE XI.—LONG-TIME TESTS USING COPPER CATALYST (1937).

Method of Sludge Determination—8 Laboratories—155 Determinations		Sludge, per cent by weight											
		7-day Test						14-day Test					
		Centrifuge		Fritted Glass Crucible		Gooch Crucible		Centrifuge		Fritted Glass Crucible		Gooch Crucible	
		Min.–Max.	Average	Min.–Max.	Average	Min.–Max.	Average	Min.–Max.	Average	Min.–Max.	Average	Min.–Max.	Average
Sludge, per cent by weight	All laboratories	0.004–0.54	0.121	0.004–0.23	0.082	0.003–0.22	0.088	0.01–0.93	0.235	0.004–0.40	0.173	0.004–0.42	0.187
	All laboratories except highest	0.004–0.20	0.070	0.004–0.14	0.065	0.003–0.21	0.072	0.01–0.32	0.148	0.004–0.32	0.145	0.004–0.30	0.158
	All laboratories except highest and lowest	0.02–0.20	0.079	0.005–0.14	0.073	0.005–0.21	0.082	0.02–0.32	0.168	0.02–0.32	0.165	0.03–0.30	0.180
Coefficient of variation	All laboratories	126		96		88		114		77		71	
	All laboratories except highest	80		97		92		76		77		71	
	All laboratories except highest and lowest	69		88		90		64		64		58	
Mean Deviation, per cent	For all laboratories (within laboratories)	9–85	38	3–63	16	4–69	13	1–54	19	3–24	20	3–57	16

Judging from the results of all laboratories, it would seem as though the centrifuging method of determining the sludge was erratic. However, by leaving out the results of the laboratory reporting the highest results, we see the centrifuging method to be as reproducible as the others. This is still more

TABLE XII.—SLUDGE ACCUMULATION TESTS USING NO CATALYST 1932–1933 (8 LABORATORIES—200 DETERMINATIONS).

Oil	21 days at 120 C.					35 days at 120 C.					42 days at 120 C.					49 days at 120 C.					56 days at 120 C.				
	Sludge, per cent by weight		Coefficient of Variation Between Laboratories	Mean Deviation Within Laboratories, per cent[a]		Sludge, per cent by weight		Coefficient of Variation Between Laboratories	Mean Deviation Within Laboratories, per cent[a]		Sludge, per cent by weight		Coefficient of Variation Between Laboratories	Mean Deviation Within Laboratories, per cen[a]		Sludge, per cent by weight		Coeffi cient of Variation Between Laboratories	Mean Deviation Within Laboratories, per cent[a]		Sludge, per cent by weight		Coefficient of Variation Between Laboratories	Mean Deviation Within Laboratories, per cent[a]	
	Min.-Max.	Average		Min.-Max.	Average	Min.-Max.	Average		Min.-Max.	Average	Min.-Max	Average		Min.-Max.	Average	Min.-Max.	Average		Min.-Max.	Average	Min.-Max.	Average		Min.-Max.	Average
A-33....	0.003-0.050	0.020	83	3–72	38	0.011–0.060	0.026	105	21–26	24	0.022–0.080	0.039	56	2–38	20	0.022–0.130	0.061	58	25–45	35	0.041–0.170	0.083	56	2-8	5
B-33....	0.005–0.080[b]	0.026	84	63–66	65	0.019–0.070[b]	0.044	40	6–21	13	0.026–0.090	0.060	31	5–12	9	0.036-0.120	0.075	35	21–26	24	0.055–0.165	0.117	32	1–4	2
C-33....	0.005–0.150	0.036	125	13–99	56	0.014–0.06	0.041	28	12–44	28	0.023–0.12	0.069	46	5–6	6	0.018–0.190	0.090	55	25–36	31	0.04–0.22	0.126	48	1–10	6
D-33....	0.07–0.42	0.177	77	18–21	20	0.14–0.35	0.265	24	3–15	9	0.17–0.42	0.326	23	1–13	7	0.25–0.51	0.399	23	0.4–4	2	0.33–0.61	0.467	21	1–11.5	6
Average..........		0.065	92	....	40	....	0.094	49	....	18	....	0.124	39	....	10	....	0.156	43	....	23	....	0.198	39	....	5

[a] The mean deviation within laboratories is for the check results of 2 laboratories only.

[b] In most cases the very high or very low results were not reproducible, as evidenced in some cases by a lower value of percentage sludge for a longer period of test.

TABLE XIII.—SLUDGE ACCUMULATION TESTS USING NO CATALYST 1931-1932 (8 LABORATORIES—168 DETERMINATIONS).

Oil	25 days at 120 C.					35 days at 120 C.					45 days at 120 C.					55 days at 120 C.				
	Sludge, per cent by weight		Coefficient of Variation Between Laboratories	Mean Deviation Within Laboratories, per cent[a]		Sludge, per cent by weight		Coefficient of Variation Between Laboratories	Mean Deviation Within Laboratories, per cent[a]		Sludge, per cent by weight		Coefficient of Variation Between Laboratories	Mean Deviation Within Laboratories, per cent[a]		Sludge, per cent by weight		Coefficient of Variation Between Laboratories	Mean Deviation Within Laboratories, per cent[a]	
	Min.-Max.	Average		Min.-Max.	Average	Min.-Max.	Average		Min.-Max.	Average	Min.-Max.	Average		Min.-Max.	Aver age	Min.-Max	Average		Min.-Max.	Average
No. 1..........	0.006–0.056	0.021	79	18–21	20	0.013–0.074	0.038	71	10–18	14	0.026–0.108	0.049	18	20–26	23	0.040–0.142	0.083	56	7–37	22
No. 2..........	0.001–0.054	0.018	94	8–55	32	0.013–0.073	0.034	53	16–37	27	0.026–0.145	0.071	49	19–43	31	0.053–0.179	0.114	37	5–28	17
No. 3..........	0.001–0.058	0.022	80	18–40	24	0.010–0.066	0.038	45	3–28	22	0.023–0.177	0.083	58	12–38	24	0.059–0.188	0.115	40	2–37	18
No. 4..........	0.077–0.259	0.147	11	10–13	11	0.137–0.322	0.212	18	1–7	5	0.181–0.448	0.305	26	2–19	10	0.255–0.699	0.469	33	1–37	15
Average..............		0.052	66	....	22	....	0.081	47	....	17	....	0.127	38	....	22	....	0.195	41	....	18

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[a] The mean deviation within laboratories is for the check results of 3 laboratories only.

evident when the erratic results, both high and low, are left out.

The Effect of the Duration of Test and the Amount of Sludge Formed on the Reproducibility of Sludge Accumulation Tests Using a Copper Catalyst:

The effect of the duration of test and the amount of sludge formed on the reproducibility of recent sludge accumulation tests (1939–1942) has been discussed above.

Table VIII, for the supplementary procedure of 1939 (air blown through the oil), shows a much lower coefficient of variation between laboratories for the 14-day test. These tests were, however, performed by only 3 laboratories, and may not be significant.

Table IX, for tests performed during 1933, shows an appreciable decrease in the coefficient of variation for the 13-day test; however, Table X, for tests performed during 1934–1935, shows no significant change of the coefficient of variation related to the duration of test.

Table XI, for tests performed during 1937, shows a somewhat lower coefficient of variation for the 14-day test than for the 7-day test.

The above results, in general, show a little better reproducibility for the 14-day test than for the 7-day test. Recent tests (1939–1942) show little difference in the reproducibility of the 3-, 7-, or 14-day tests.

SLUDGE ACCUMULATION TESTS USING NO CATALYST

General:

This sludge accumulation test is the same as the sludge accumulation tests described above, except that no catalyst is used. It also differs from the recent tests in that the air supply is not dried, and the sludge is determined by centrifuging.

Calculated Results:

The data for Tables XII and XIII are taken from the October 20, 1933 report of Section B. Table XII is for tests of 1931–1933 and Table XIII for tests of 1931–1932. (In both of these tables results of the Detroit Edison Laboratory have been omitted because of wide divergence from the results of other laboratories.)

The Effect of the Duration of the Test and the Amount of Sludge Formed on the Reproducibility of Sludge Accumulation Tests Without Copper Catalyst:

From Table XIV, it is seen that the coefficient of variation between laboratories of sludge accumulation tests using no copper catalyst varied from 11 per cent to 125 per cent with an average coefficient of variation of 50 per cent. These values for the coefficient of variation are for sludge ranges of 0.02 per cent to 0.47 per cent, and 21 to 56 days of test. From Tables XII and XIII, it is seen that, in general, the coefficient of variation decreases as the days of test increase. However, after a period of about forty days, the coefficient of variation levels off at approximately 40 per cent.

TABLE XIV.—SUMMARY OF SLUDGE ACCUMULATION TESTS, NO COPPER CATALYST (1931–1933).

Summary of Tables XII and XIII (8 Laboratories—368 Determinations).

Days of Test	Sludge, per cent by weight, min.–max.	Coefficient of Variation Between Laboratories, per cent		Average Mean Deviation within a Laboratory, per cent	
		Min.–Max.	Average	Min.–Max.	Average
21–56	0.02–0.47	11–125	50	6–40	19

Comparison of the Reproducibility of Sludge Accumulation Tests With and Without Copper Catalyst:

From Table VII, it is seen that the coefficient of variation for recent sludge accumulation tests using copper catalyst, varies from 20 per cent to 51 per cent with an average value of 35 per cent.

By a comparison of Tables VII and XIV, it appears that the results of the recent tests using copper catalyst are somewhat more reproducible within each individual laboratory, than are the results of tests using no catalyst, the average percentage mean deviation of the check results run within a laboratory is about 8 per cent. The corresponding figure for all tests using no catalyst is 19 per cent and 16 per cent when considering only test days of 42 to 56 days. However, it should be kept in mind that the percentage mean deviation for tests using no catalyst are for fewer tests than for the tests using copper catalyst, and thus may not be so representative of actual conditions.

These results indicate that the recent sludge accumulation tests using copper catalyst are somewhat more reproducible than the tests using no catalyst, 1931–1933. However, these tests using copper as a catalyst are of a later date than those using no catalyst, and due to refinements of technique, the later tests might yield lower deviations at the present time. This is evidenced by the greater coefficient of variation for early sludge accumulation tests using copper catalyst, which were performed about the same period of time as the tests using no catalyst.

The Reproducibility of the Sludge Measuring Procedure:

Cooperative tests were performed in 1932 to determine the effect of the sludge content of an oxidized oil on the reproducibility of the sludge measuring procedure. The procedure used was that described in the circular letter of August 1932 sent out by Subcommittee IV, and was one employing precipitation of the sludge by A.S.T.M. naphtha, followed by centrifuging and weighing; the tests were performed on samples of oils previously oxidized. The data for Table XV are taken from the November 1, 1932 report of Section B.

By comparing Table XV with Tables XII, XIII, and XIV (sludge accumulation tests using this sludge-measuring procedure) it is seen that only a small portion of the deviations of the results is due to the error involved in measuring the sludge. From Table XI, it is seen that all other conditions being equal, the coefficient of variation is not greatly affected by using a Gooch or fritted glass crucible in place of the centrifuge. Therefore, it may be said that the greater portion of the deviations arises from sources other than the sludge-measuring procedure, and is due to persistent differences of technique from one laboratory to another.

TABLE XV.—REPRODUCIBILITY OF THE SLUDGE MEASURING PROCEDURE (11 LABORATORIES, 72 DETERMINATIONS)

Oil	Sludge, per cent by weight		Coefficient of Variation Between Laboratories	Mean Deviation Within a Laboratory, per cent	
	Min.-Max.	Average		Min.-Max.	Average
No. 1...	0.335–0.382	0.354	7	0.6– 1.5	0.9
No. 2...	0.145–0.160	0.151	3	0 – 3.3	1.2
No. 3...	0.045–0.061	0.052	9	0.9–11.1	4.4
Average		0.19	6		2

THE REPRODUCIBILITY OF THE SNYDER LIFE TEST

The Snyder life test measures the time, in days, for initial traces of sludge deposition under controlled conditions. The oil is contained in a beaker which is kept in an oven at 120 C. Oil samples are removed periodically and then centrifuged, and any sludge deposition is noted. The value of sludge life is taken as the number of days for initial sludge deposition.

In Tables XVI and XVII, oils with a Snyder life test of one day or less have been omitted.

Summary of all Snyder Life Tests (Tables IX, X, XI).—The oils may be divided into three groups, according to their sludge life range, as follows:

Oils	Min.-Max. Sludge Life, days
8, 10, 27-2	2–9
2, 3, 4, 5, 6, 7, 9, 11, 27-3, A-1, B-2, B-3, C-1, C-2, R-2, R-3	12–30
12	76

Eight laboratories cooperated on each series of tests.

TABLE XVI.—A.S.T.M. TESTS OF 1926 (REPORT OF SECTION B, DECEMBER 29, 1926) (8 LABORATORIES—190 DETERMINATIONS).

Oil	Sludge Life, days		Coefficient of Variation Between Laboratories, per cent	Mean Deviation Within Laboratories, per cent	
	Min.–Max.	Average		Min.–Max.	Average
25–2	11–25	17	26	0–10	4
25–3	11–23	16	27	0–13	3
25–4	11–21	17	22	0–24	10
25–5	11–19	14	23	0–30	9
25–6	14–27	19	20	0–15	3
25–7	50–113	76	8	1–7	4
25-8	5–19	9	46	0–39	8
25–9	9–15	12	15	0–12	4
25–10	4–16	9	44	0–6	2
25–11	10–24	16	30	0–26	8
25–12	15–39	26	17	0–7	4
25–14	13–21	16	..	0–4	2

Most of the oils tested fell within the group 12–30 days life test, and had an average coefficient of variation of 22 per cent. It can be seen from Table XIX that, in general, the coefficient of variation decreases as the number of days of test increases. This is to be expected, since the percentage error involved in estimating the number of days for the initial sludge deposition decreases as the number of days of test increases, due to the decreasing importance of a day more or less, in relation to the time taken.

The Snyder life test is somewhat more reproducible than the sludge accumulation tests, having an average coefficient of variation of 25 per cent. This compares with 35 per cent for sludge accumulation tests using copper catalyst (1939–1942) and 40 per cent for sludge accumulation tests without catalyst (tests of 1931–1933, for test periods *greater than 40 days*).

TABLE XVII.—INTERLABORATORY TESTS BY I.E.C. TRANSFORMER OIL ADVISORS (1927) (8 LABORATORIES—112 DETERMINATIONS).

Oil	Sludge Life, days		Coefficient of Variation Between Laboratories, per cent	Mean Deviation Within Laboratory, per cent	
	Min.–Max.	Average		Min.–Max.	Average
A-1	21–48	29	31	1–18	7
B-2	7–19	13	27	0–14	5
B-3	18–48	30	22	1–20	7
C-1	12–19	16	18	0–20	4
C-2	9–18	15	21	0–29	8
R-2	10–18	14	20	0–8	5
R-3	20–44	28	26	0–18	4

TABLE XVIII.—A.S.T.M. TESTS ON "27" SERIES OILS (REPORT OF SECTION B—JUNE 28, 1927) (8 LABORATORIES—28 DETERMINATIONS).

Oil	Sludge Life, days		Coefficient of Variation Between Laboratories, per cent	Mean Deviation Within Laboratories, per cent	
	Min.–Max.	Average		Min.–Max.	Average
27-2	1– 3	2	44	0–50	18
27-3	13–16	15	10	0–32	2

TABLE XIX.—SUMMARY OF ALL SNYDER LIFE TESTS (330 DETERMINATIONS).

Number of Oils	Average Sludge Life, days		Coefficient of Variation Between Laboratories, per cent		Average Mean Deviation Within Laboratories, per cent	
	Min.–Max.	Average	Min.–Max.	Average	Min.–Max.	Average
3	2–9	7	44–46	45	2–18	9
16	12–30	19	10–31	22	2–10	6
1		76		8		4
Weighted Average	2–76		8–46	25	2–18	6

THE REPRODUCIBILITY OF THE SLIGH SHORT-TIME OXIDATION TEST

The Sligh test was developed by the National Bureau of Standards as a measure of the oxidation tendency of an oil. A weighed sample of the oil is oxidized in a closed flask in an atmosphere of oxygen (at atmospheric pressure) for $2\frac{1}{2}$ hr. at 200 C. The sludge is precipitated with A.S.T.M. naphtha, and the oxidized oil filtered, using a Gooch crucible. The weight of this precipitate, in milligrams per 10 g. of sample, is the Sligh oxidation number of the oil.

TABLE XX.—A.S.T.M. TESTS OF 1935 (REPORT OF SUBCOMMITTEE IV, JUNE 18, 1935) (4 LABORATORIES—16 DETERMINATIONS.

Oil	Sligh Oxidation Number		Coefficient of Variation Between Laboratories, per cent
	Min.-Max.	Average	
A-34	5.1- 9.4	6.9	22
C-34	12.0-14.6	12.7	14
B-34	14.5-18.8	16.5	9
D-34	31.7-36.4	33.6	7
Average	5.1-36.4	17.4	13

TABLE XXI.—A.S.T.M. TESTS OF 1932 (REPORT OF SUBCOMMITTEE IV, JUNE 13, 1932) (8 LABORATORIES—48 DETERMINATIONS).

Oil	Sligh Oxidation Number		Coefficient of Variation Between Laboratories, per cent	Mean Deviation Within Laboratories, per cent[a]	
	Min.-Max.	Average		Min.-Max.	Average
1	6.7-10.2	8.4	15	3.0-3.4	3.4
3	11.7-18.7	15.3	15	6.3-7.2	6.8
2	14.1-24.2	19.6	13	3.5-5.7	4.6
4	28.6-48.8	38.2	16	2.4-2.5	2.5
Average		20.4	15	2.4-7.2	4

[a] The mean deviation is for the check results of two laboratories only.

It can be seen from Tables XX to XXIII that, in general, the coefficient of variation is lower for the higher sludging oils. The coefficient of variation for Sligh oxidation tests of 1931–1935 varied from 6 to 22 per cent, with an average value of 12 per cent for sligh oxidation numbers of 6.9 to 38.2 (corresponding to 0.069 + 0.382 per cent sludge by weight).

The Sligh oxidation test is thus seen to be quite reproducible, and is the most reproducible of all the tests covered in this report.

TABLE XXII.—A.S.T.M. TESTS OF 1931 (REPORT OF SUBCOMMITTEE IV, JUNE 17, 1931) (6 LABORATORIES—18 DETERMINATIONS).

Oil	Sligh Oxidation Number		Coefficient of Variation Between Laboratories, per cent
	Min.-Max.	Average	
C-1	22.3-32.5	27.4	11
C-2	22.9-30.5	27.8	6
C-3	25.9-33.5	31.6	6
Average		29.9	8

TABLE XXIII.—SUMMARY OF SLIGH OXIDATION TESTS (1931-1935).

Summary of Tables XX, XXI and XXII (4 to 8 Laboratories—82 Determinations).

Oils	Number of Oils	Average Sligh Oxidation Number		Coefficient of Variation Between Laboratories, per cent	
		Min.-Max.	Average	Min.-Max.	Average
A-34, 1	2	6.9- 8.9	7.7	15-22	18
C-34, B-34, 2, 3	4	12.7-19.6	16.0	9-15	13
D-34, 4, C-1, C-2, C-3	5	27.4-38.2	31.7	6-16	9
Weighted Average		6.9-38.2	21.6	6-22	12

THE REPRODUCIBILITY OF THE HIGH PRESSURE OXIDATION TEST

General:

The high pressure oxidation test is, essentially, a measure of the oxidation tendency of an oil accelerated by a temperature of 140 C. and oxygen at 250 psi. pressure. After 24 hr. of this accelerated oxidation, the sludge is precipitated by A.S.T.M. naphtha, and the oxidized oil filtered, using a Gooch crucible. The "Bomb Sludge Value" is the weight per cent of sludge formed.

TABLE XXIV.—HIGH-PRESSURE OXIDATION TESTS (1939–1941).

Oil[a]	Sludge, per cent by weight		Coefficient of Variation Between Laboratories, per cent	Mean Deviation Within a Laboratory, per cent	
	Min.-Max.	Average		Min.-Max.	Average
TESTS OF 1940–1941, 9 LABORATORIES—39 DETERMINATIONS					
A....	0.014–0.029	0.019	24	0. –10	5
B....	0.075–0.128	0.094	18	2.6– 3.9	3
Average		0.06	21		4
TESTS OF 1939–1940, 10 LABORATORIES—45 DETERMINATIONS					
B....	0.009–0.017	0.014	29	0–18	5
A....	0.025–0.060	0.043	27	0.1–19	7
Average		0.03	28		6
TESTS OF 1939, 6 LABORATORIES—30 DETERMINATIONS					
B....	0.013–0.034	0.019	37	6–18	10
A....	0.037–0.079	0.053	16	2–20	7
Average		0.04	27		9
Grand Average, 1939 to 1941...			25		6

[a] The designation of the oil as A orB does not have any special significance and the same oils were not tested in each series of tests.

Calculated Results:

The data for Table XXIV are taken from reports of Section B. Data for tests of 1940–1941 are taken from the June 20, 1941 report, for tests of 1939–1940 from the January 9, 1940 report, and for tests of 1939 from the October 18, 1939 report.

The Effect of the Amount of Sludge Formed on the Reproducibility:

From Table XXIV it is seen that for each series of tests, the tests on the higher sludging oil had a better reproducibility.

High Pressure Oxidation Tests of 1939–1941 Compared with Other Methods of Test:

The high pressure oxidation tests of 1939–1941 had a coefficient of variation ranging from 16 per cent to 37 per cent with an average value of 25 per cent. The latest tests of 1940–1941 had a coefficient of variation somewhat lower than tests of 1939–1940 and 1939, indicating some improvement, with the acquisition of experience with the test, by the persons carrying out the work.

The average value of 25 per cent for the coefficient of variation of high pressure oxidation tests compares with 12 per cent for the Sligh test, 25 per cent for the Snyder life test, 35 per cent for sludge accumulation tests using copper catalyst of 1939–1942 (latest tests of 1942 had a coefficient of variation of 27 per cent, and 50 per cent for sludge accumulation tests without catalyst of 1931–1933 (40 per cent for periods of test greater than 40 days).

THE REPRODUCIBILITY OF MODIFIED SLIGH TESTS

In an effort to reduce the high temperature required in the Sligh oxidation

TABLE XXV.—REPRODUCIBILITY OF MODIFIED SLIGH TESTS, 1931.

Oil	150 C., 4 hr., 5 Laboratories, 15 Determinations			120 C., 4 hr., Copper Catalyst, 4 Laboratories, 12 Determinations			100 C., 6 hr., Copper Catalyst, 4 Laboratories, 12 Determinations		
	Sligh Oxidation Number		Coefficient of Variation Between Laboratories, per cent	Sligh Oxidation Number		Coefficient of Variation Between Laboratories, per cent	Sligh Oxidation Number		Coefficient of Variation Between Laboratories, per cent
	Min.-Max.	Average		Min.-Max.	Average		Min.-Max.	Average	
C-1...........	0–1.1	0.71	62	0–1.8	0.7	96	0–1.2	0.7	89
C-2...........	0–2.2	1.20	74	0–2.1	0.9	92	0–1.1	0.75	58
C-3...........	2.3–3.6	2.90	22	1.5–2.8	2.1	23	0–1.9	1.25	60
Average		1.6	53		1.2	70		0.9	69

test method, tests were conducted in 1931 to determine the effect of lower temperatures, more time, and copper catalyst.

Table XXV is calculated from data of the June 17, 1931 report of Subcommittee IV.

These values of the coefficient of variation are rather high, but would be reduced by more time under the given conditions.

TABLE XXVI.—REGULAR SLIGH TEST—IDENTICAL ALUMINUM OVEN (3 LABORATORIES—32 DETERMINATIONS.

Oil	Sligh Oxidation Number		Coefficient of Variation Between Laboratories, per cent	Mean Deviation Within a Laboratory, per cent	
	Min.-Max.	Average		Min.-Max.	Average
1....	9.6-13.2	10.8	16	0.4-3.4	2
2....	21.2-23.5	18.8	6	3.4-5.6	5
3....	18.4-19.4	22.3	4	3.3-6.9	5
4....	33.9-39.7	37.4	7	4.0-5.4	5
Average.............		22.3	8		4

Tests were performed in 1932 using the regular Sligh test, but employing the identical aluminum oven as the heating element. These tests were performed by three laboratories only. (Data from report of Section B, June 13, 1932.)

The advantages of using exactly the same type of oven are seen by comparing Tables XXVI and XXI. These tests were carried out on the same oils, and the coefficient of variation for all laboratories was 15 per cent, and only 8 per cent for those three laboratories using the identical aluminum block oven.

TABLE XXVII.—COMPARISON OF THE REPRODUCIBILITY OF COOPERATIVE DETERMINATIONS OF THE OXIDATION TENDENCY OF OILS (1926-1942).

Method of Test	Test Years	Average Per Cent Sludge by Weight	Coefficient of Variation Between Laboratories, per cent	
		Min.-Max.	Min.-Max.	Average
Recent Sludge Accumulation Tests Using Copper Catalyst	1939-1942	0.04 -0.66	20-51	35
Other Sludge Accumulation Tests Using Copper Catalyst	1933-1939			
Supplementary Procedure:				
Air blown through the oil	1939	0.15 -0.25	6-39	23
Oxidized at 120 C...........	1933-1935	0.009-0.98	21-120	61
Sludge Accumulation Tests Using No Catalyst..........	1931-1933	0.02 -0.47	11-125	50
		Sludge Life, days		
Snyder Life Test	1926-1927	2-76	10-46	25
		Sligh Oxidation Number		
Sligh Test........	1931-1935	6.9-38.2	6-22	12
		Sludge, per cent by weight		
High Pressure Oxidation Test...	1939-1941	0.014-0.094	16-37	25
		Sligh Oxidation Number		
Modified Sligh Tests. Regular Sligh Test, but Using Identical Aluminum Oven..........	1932	10.0-37.4	7-16	8
150 C., 4 hr....	1931	0.7- 2.9	22-74	53
120 C., 4 hr., Copper Catalyst...........	1931	0.7- 2.1	23-96	70
100 C., 6 hr., copper catalyst...........	1931	0.7- 1.3	58-89	69

MECHANICAL TESTS OF CELLULOSE ACETATE—PART II ON CREEP

By William N. Findley[1]

Synopsis

Creep properties of cellulose acetate as obtained by tests at several different stresses are presented. All tests were conducted in a constant-temperature, constant-relative-humidity laboratory on specimens obtained from the same sheet of material used for the tests reported in 1941.[2] Creep readings were taken for periods of time up to 10 months. The data are analyzed to show the effect of stress on the rate of creep, the effect of stress on the creep for a given time interval, the effect of aging of the material on the rate of creep, and the effect of aging on the time for fracture.

It is known that many plastics undergo considerable deformation over a period of time as a result of sustained loads of relatively small magnitude. This deformation may continue until fracture takes place if the loads are maintained a sufficient length of time.

In a previous paper the effect of stress on the time required to cause fracture of cellulose acetate under a constant applied load was shown.[2] It was found that fracture took place after a period of time even for stresses far below the ultimate strength as determined from a short-time tension test.

The present tests are a continuation of the tests mentioned above. The tests reported herein concern the effect of time on the deformation of cellulose acetate when subjected to a constant tensile load for periods of time up to 7000 hr. (about 10 months) and at various stresses—the phenomenon of "creep." Two other investigators have studied this phenomenon for different types of loading. Bartoe[3] has studied the effect of a steady compressive load on methyl methacrylate for periods of time up to 1000 hr. Delmonte and Dewar[4] have studied the effect of a steady bending moment on several plastics for periods of time up to 100 hr. at various temperatures but at only one stress, 1000 psi.

Material

The material[5] for these tests was supplied by the Plastics Division of the Monsanto Chemical Co. (Monsanto formulation number 2050TV). All specimens used in these tests were cut from the same sheet of cellulose acetate as used for the 1941 tests.

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[1] Instructor in Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[2] William N. Findley, "Mechanical Tests of Cellulose Acetate," *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 1231 (1941).

[3] W. F. Bartoe, "Service Temperature Flow Characteristics of Thermoplastics," *Mechanical Engineering*, Vol. 61, No. 12, December, 1939, p. 892.

[4] J. Delmonte and W. Dewar, "Factors Influencing Creep and Cold Flow of Plastics," ASTM Bulletin, No. 112, October, 1941, p. 35.

[5] For complete description of the material, see 1941 paper referred to in footnote 2.

SPECIMENS

A drawing of the specimen used for these creep tests is shown in Fig. 1. The specimens were cut from the sheet of cellulose acetate with the same orientation as the specimens used in the previous tests, namely, with their axes crosswise of the sheet. All specimens were cut on a jig saw and then milled to the form shown in Fig. 1. The milled edges were then smoothed with No. 00 emery paper so as to remove all burrs and scratches transverse to the axis of the specimen. All specimens used for the creep tests were prepared at the same time, placed in the laboratory at the same time, and remained in the laboratory continuously thereafter. The laboratory was maintained at a constant temperature of 77 ± 1 F., and 50 ± 2 per cent relative humidity continuously throughout the duration of the tests, about 10 months.

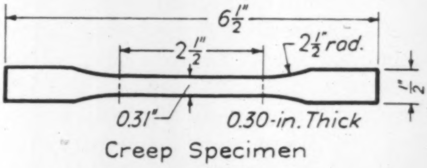

FIG. 1.—Creep Specimen.

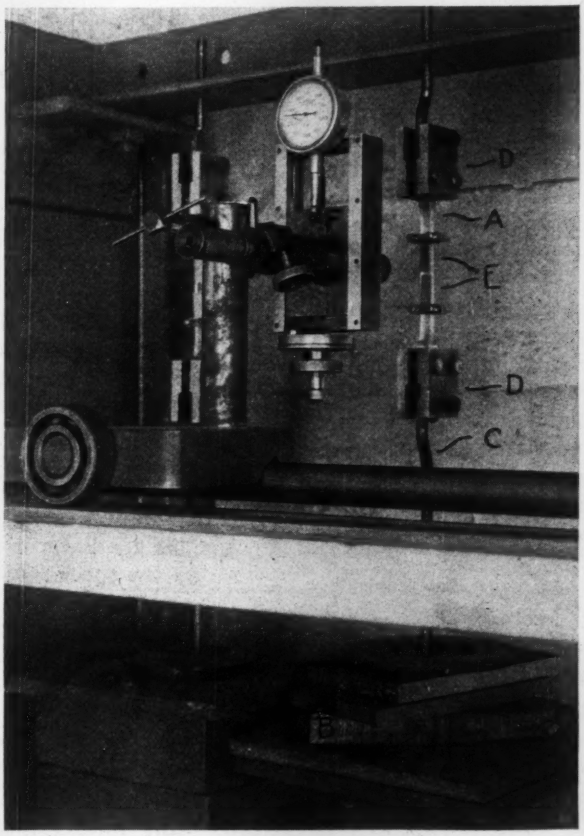

FIG. 2.—Creep Testing Equipment.

TESTING EQUIPMENT

The equipment used for conducting the tests consisted of a steel rack from which 24 specimens could be suspended, calibrated weights used for loading the specimens, measuring equipment for determining the strain in each specimen, and a clock equipped with a counter to record the elapsed time in hours.

Because of the sensitivity of cellulose acetate to small changes in temperature and relative humidity, it was also necessary to provide a test room maintained at constant temperature and constant relative humidity throughout the tests.

Figure 2 shows a portion of the creep rack with apparatus set up for measuring the strain of a specimen. As may be seen the specimen, *A*, was subjected to an axial tensile load by means of dead weights, *B*, attached to the rod *C*. The specimen was held by grips, *D*, which contained a hook-and-eye type of swivel joint. This joint was provided in order to minimize the possibility of bending the specimen.

The extensometer used for measuring the creep consisted of two T-shaped brass strips, *E*, clamped to the specimen with a traveling microscope (cathatometer, *F*) for measuring the displacement between fixed reference marks on the ends of the two brass strips. A track was provided for the microscope so that it could be moved from specimen to specimen quickly.

The brass strips *E* were clamped to the specimen with compressed springs. These springs were necessary in order to keep the extensometer firmly attached to the specimen during the test, since a reduction of area of 30 per cent might be expected before fracture took place.

The distance between the centers of the flat bars, to which the above-mentioned springs were attached, was considered to be the gage length of the extensometer. This distance was 2 in. at the start of the test. A uniform gage length for each specimen was essential for accurate comparison of tests. This uniformity was obtained by using identical extensometers and a jig to assemble the extensometer to the specimen.

Flat clamps were used to attach the extensometers to the specimens instead of pointed screws because creep of the material would cause screws to sink into the specimen, this causing early failure. The flat clamps were found to yield a satisfactorily precise location of gage length inasmuch as it was found that slippage between the clamps and the specimen as a result of the stretching of the specimen was distributed equally on each side of the clamp bar.

PROCEDURE

Twelve specimens were prepared as outlined above and allowed to remain in the atmosphere of the testing room for about 2 weeks before any tests were started. Tests were started with eight of the specimens at the same time but with different stresses ranging from 505 to 2695 psi. The procedure in starting these tests was as follows:

The weights, weighing from 30 to 150 lb., were first supported on planks, blocked up in such a way that they could be used as levers to lower the weights quickly but gently until they were supported by the specimen. Before lowering the weights, the initial extensometer readings were obtained with the traveling microscope. Then the weight was lowered on the first specimen, the extensometer was immediately read again, and the time was recorded. The difference between the strain computed from these two sets of readings was the elastic strain. Any further increase in strain was the result of creep. The remaining seven specimens were treated in the same way.

After all eight tests had been started, strain and time readings were taken at intervals of time, which for the high stress tests were from 2 to 12-hr. intervals until fracture. Low stress specimens were read about every 2 days for a month, then every 1 to 2 weeks.

All specimens were weighed both before the start of the test and after fracture to ascertain any change in weight.

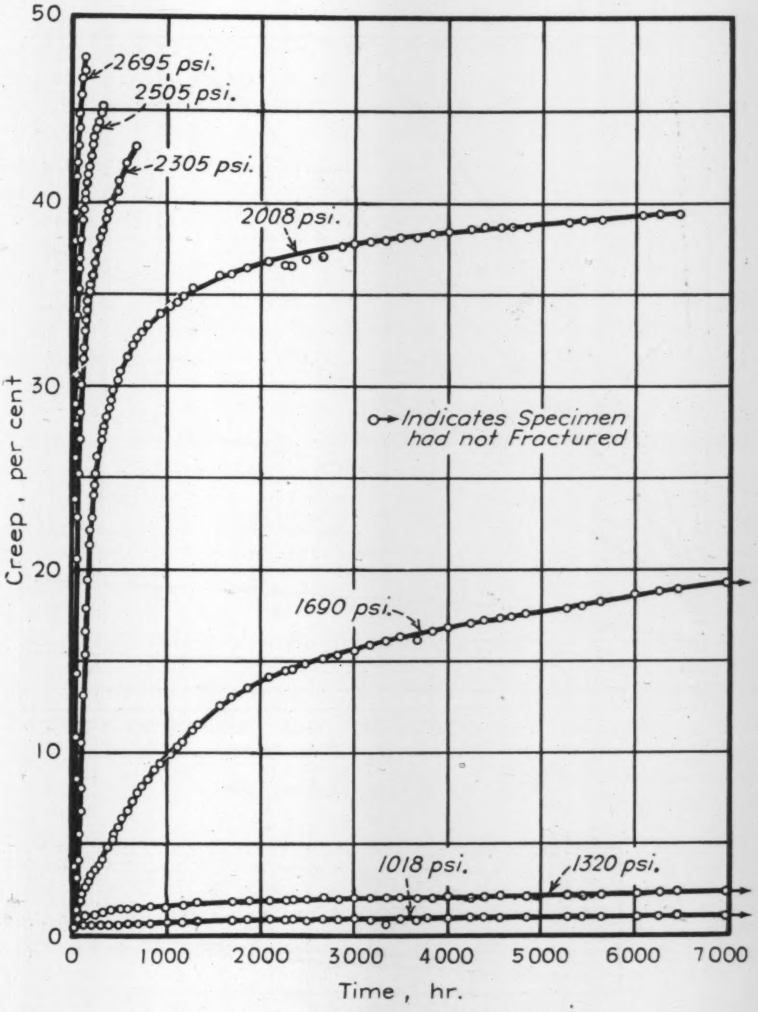

FIG. 3.—Creep-Time Diagrams, 7000 hr.

TEST RESULTS

The results of seven of these eight tests are shown in Fig. 3 where creep is plotted against time. Creep as usually defined is the total change in length (including elastic stretch) between gage points, located on the cylindrical portion of the specimen, expressed as a percentage of the original distance between gage points. For the sake of clarity the test data at 505 psi. stress were omitted from Fig. 3 but are shown for the first 2000 hr. in Fig. 4. However, there was no measurable creep at this stress.

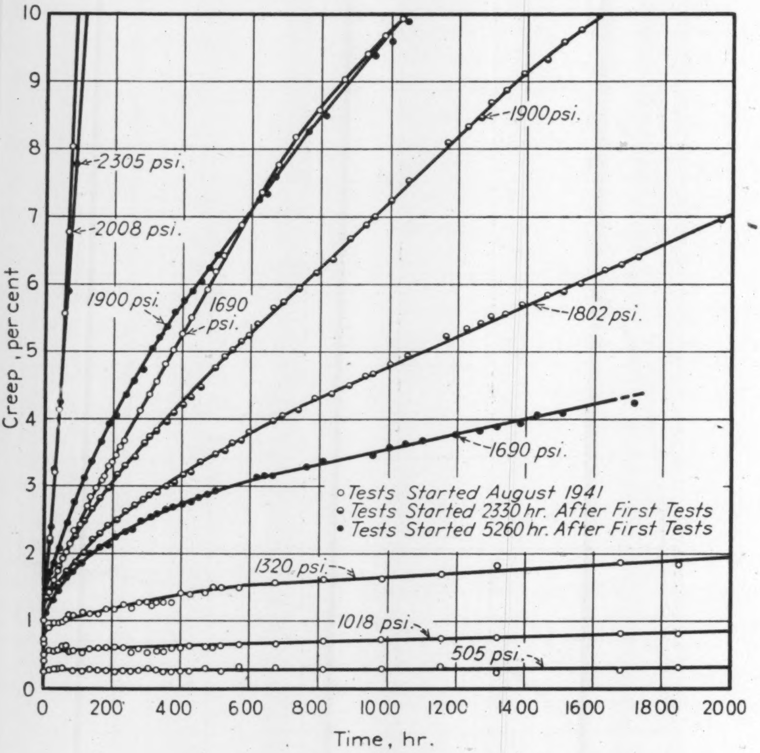

FIG. 4.—Creep-Time Diagrams, 2000 hr.

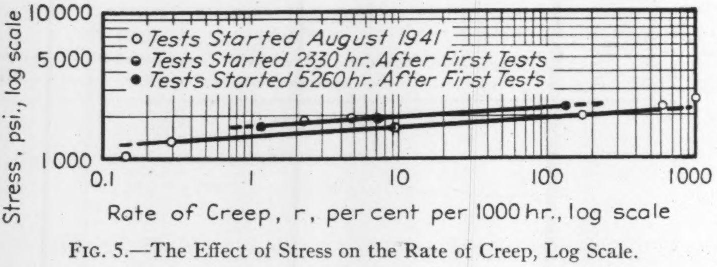

FIG. 5.—The Effect of Stress on the Rate of Creep, Log Scale.

From Fig. 3 it is evident that the intermediate stresses (2008 and 1690 psi.) resulted in an initial rapid rate of creep at a constant rate (referred to here as the first stage of creep) followed by a transition region (referred to here as the second transition) and then another constant rate of creep (referred to here as the second stage of creep). It is evident from Fig. 3 that the rate of creep is much slower in the second stage than it is in the first stage. An examination of Fig. 3 shows that the second stage is practically nonexistent at the higher stresses (2305, 2505, 2695 psi.). Also for the low stresses (1018 and 1320 psi.) the second stage either is nonexistent or had not yet been reached after 7000 hr. of testing.

The data for the first 2000 hr. are shown to a larger scale in Fig. 4. In this diagram the data from the original set of eight specimens are shown by open circles. It is evident from this plot that there is an initial or first transition region preceding the first stage of creep. The curvature of this transition is concave downward for intermediate and low stresses and concave upward for the high stresses.

It will be noticed that no final region of increasing rate of creep occurred, such as the "third stage" of creep in metals. This is probably due to the fact that there is no localized reduction of area (necking down) just before fracture of this material, as there is for metals.

Effect of Aging:

After an elapse of 2330 hr. (about 3 months) from the start of these tests two more specimens were started at 1802 and 1900 psi. in order to obtain more information at intermediate stresses. The resulting data are shown by the half filled circles in Fig. 4. It may be seen that the rate of creep of these specimens is much lower than was to have been expected on the basis of the first tests. For example the data for both tests of this second group (1802 and 1900 psi.) fall below the data for the 1690 psi. test of the first group. Also it may be seen that the character of the initial transition has changed slightly from that observed in the first set of tests.

The differences in behavior of these two sets of tests can only be the result of the elapse of time between the start of the first tests and that of the second tests (aging) because all other conditions were held constant. All specimens were made at the same time from the same sheet of acetate, and had been in the air-conditioned laboratory the same time. By aging is meant a change in the mechanical properties of the material with time. Possible causes might be: slow change in moisture content; residual solvent content or plasticizer; or perhaps further polymerization.

In order to study this phenomenon futher, tests of the remaining two specimens were started 5260 hr. after the start of the first test, at stresses of 1690 and 1900 psi. The specimen used for the first test at 505 psi. was also changed to a stress of 2305 psi. (The specimen had not shown any creep at the 505 psi. stress.) The data for these tests are represented by the solid circles in Fig. 4.

It will be noticed that the rate of creep for these tests is much less than that for the first group but is greater than the rate of creep for the second group of specimens. No explanation can be offered for this apparent reversal in the effect of aging and a further study of the effect is not possible because of a lack of specimens which have had a parallel history.

Weight measurements of specimens of the acetate show a decrease of about 0.6 per cent in weight during the period of creep testing, about 7000 hr.

Effect of Stress:

The effect of stress on the creep of the cellulose acetate is better shown by Figs. 5 and 6.[6,7] Figure 5 shows a double logarithmic plotting of stress against rate of creep (for the first stage of creep). The rate of creep is obtained as the slope of the curves shown in Fig. 3 or Fig. 4 for the fiirst stage, as defined above. It will be noticed that the data for the first group of tests, shown by the open circles, fall on a straight line, except at the two extremes where measurement of the rate of creep was uncertain. The data for the second and third group of specimens are also shown by half-filled and filled circles, respectively. These data also fall on straight lines which are parallel to the first.

Data, such as these, which yield a straight line on double logarithmic plotting may be represented by an equation of the form

$$S = m \cdot r^n$$

where:

S = the stress,

r = the rate of creep,

n = the geometric slope of the line, and

m = the value of the stress corresponding to a rate of creep of one.

This equation for the first group of tests is $S = 1440\, r^{0.065}$.

An examination of Fig. 5 shows that aging changes the constant in the above equation but does not change the exponent.

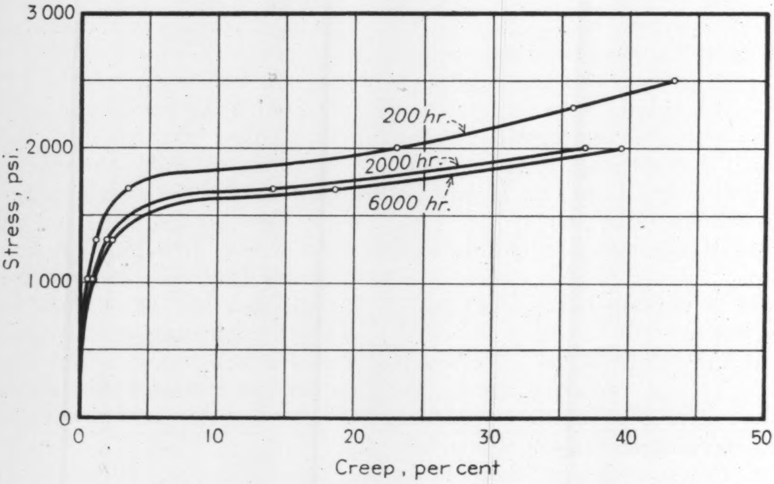

FIG. 6.—The Effect of Stress on the Creep for Three Different Time Intervals.

In Fig. 6, stress is plotted against the creep occurring in a certain time interval. Curves for time intervals of 200, 2000, and 6000 hr. are shown. It may be seen that there is a critical value of stress at about 1500 psi. below which the creep is relatively small and above which the creep is relatively large.

If it be assumed for purposes of analysis that a creep of 1 per cent in a time of 6000 hr. (about 8 months) is the limiting value above which structural damage may be said to occur for plastics, then a creep limit may be defined. It would be: The maximum stress that

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[6] P. G. McVetty, "The Interpretation of Creep Tests," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 105 (1934).

[7] H. F. Moore, "Materials of Engineering," Sixth Edition, p. 37, McGraw-Hill Book Co., Inc., New York, N. Y., 1941.

can be continuously applied at a constant temperature and relative humidity for a period of 6000 hr. without causing creep of more than 1 per cent. Under this definition the creep limit for the cellulose acetate as obtained from the first group of tests is 1000 psi. at 77 F. and 50 per cent relative humidity.

Since a practical creep limit has not yet been defined for plastics, the above value is by way of illustration only. It seems likely that different definitions of creep limit will be necessary for plastics used in different classes of service.

Effect of Time:

The effect of time of loading on the stress which would cause fracture to take place is shown in Fig. 7. In this figure stress is plotted against the time required to cause fracture at the particular stress. Double logarithmic plotting is used. The open circles represent the data for long-time fracture tests started in November, 1940, and reported in the previous paper. The filled circles represent data taken from the creep tests shown in Fig. 3. The latter tests were started August, 1941. An examination of Fig. 7 shows that the time for fracture is much less for the 1941 tests than for the 1940 tests. For example at a stress of 2400 psi. the time for fracture dropped from 1500 hr. to 500 hr.

Thus aging has reduced the time for fracture at a given stress but aging, as shown above, also decreases the rate of creep at a given stress. It should, however, be mentioned that the duration of aging was about three times as long for the two sets of fracture tests as for the first two sets of creep tests. Also the aging did not occur over the same period of time for the two groups of tests.

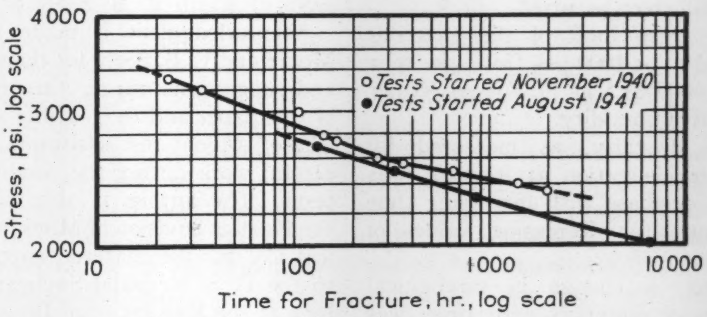

FIG. 7.—The Effect of Stress on the Time for Fracture Under Constant Load.

The effect of time of loading on the ductility of the material, as measured by percentage elongation at fracture, may be seen in Fig. 3. The percentage elongation at fracture decreases rapidly at first and then more slowly, as the time for fracture increases. For a 200-hr. test, the percentage elongation at fracture was 48 per cent. For a 6600-hr. test, the percentage elongation at fracture was 39.5 per cent.

CONCLUSIONS

The following conclusions may be drawn from the creep tests of cellulose acetate as obtained from tests extending over 10 months' time at a constant temperature of 77 F. and relative humidity of 50 per cent.

1. Two regions of constant rate of creep were observed with an initial and

an intermediate transition region of decreasing rate of creep. No final stage of rapidly increasing rate of creep occurred, such as the "third stage" of creep in metals.

2. The relationship between the stress and the rate of creep in the "first stage" of creep was found to be expressed by an equation of the form $S = 1440\ r^{0.065}$.

3. A critical value of stress was observed above which relatively large creep occurred and below which relatively small creep occurred.

4. The creep limit of this material was found to be 1000 psi. (for 1 per cent creep in 6000 hr.) at 77 F. and 50 per cent relative humidity.

5. The ductility, as measured by percentage elongation at fracture, was found to decrease with increasing time for fracture (or decreasing values of stress).

6. Aging, a change in mechanical properties of materials with time, was observed. For the acetate tested, aging caused a marked decrease in the first-stage rate of creep. It was also observed, by comparison with previously reported data, that the time for fracture under a constant load decreased with aging of the material.

Acknowledgements:

The tests reported are a part of the work of the Engineering Experiment Station of the University of Illinois, M. L. Enger, Director, in the Department of Theoretical and Applied Mechanics of which F. B. Seely is head.

Acknowledgment is made to H. F. Moore and F. B. Seely for their criticism and suggestions during the preparation of this paper and to O. Hintz and R. V. Chase, student test assistants, for their careful work during the conduct of the tests. The author is also indebted to the Plastics Division of Monsanto Chemical Co. for the material supplied, and to the U. S. Regional Soybean Laboratory at the University of Illinois for the loan of certain equipment.

DISCUSSION

Mr. John Delmonte.[1]—Mr. Findley has added a very useful set of data to our rather limited knowledge of creep phenomena.

I should like to make one or two exceptions to the points raised in the paper, and those have to do primarily with the observation of creep at very low stresses. It has been our previous experience when making measurements through use of the beam that creep phenomena was observable at stresses even under 500 psi.; we observed creep even as low as 50 to 100 psi. However, differences in measurements might be due to the technique in Mr. Findley's method *versus* our methods.

I should like to make this further observation: In making initial observations on creep phenoma I should like to know how long a period of time was required to make the first reading. For example, we find true elastic phenomena usually was measured over an instantaneous period of time. That is to say, the deformation within the first fraction of a second when the load was applied constitutes elastic phenomena; any other deformation is necessarily plastic in nature and, consequently, bears more on the subject of creep. Hence, the time to make the first observation of deformation would be quite significant.

Mr. E. B. Cooper.[2]—I should like to call to the attention of the Society's Committee D-20 on Plastics a point in connection with creep which rather recently was called to my attention by research workers in textiles. The Russians have a name for this preliminary or first deformation which I think is pretty good. The Russians call this initial deformation "acoustical deformation," because a sound wave is the most rapid mechanical pulse which can travel through a specimen. Then the textile people call the thing, which I believe we are calling creep, "delayed elasticity," from the standpoint that a great deal of it may be returned, may be gotten back over a period of time; and, finally, the textile people refer to a third item as "permanent set," and "permanent set," I am told, may be entirely eliminated by mechanical conditioning, that is, if a fiber has been "permanently set" once, the next time it shows only acoustical deformation plus delayed elasticity.

In seems to me that in our creep studies some attention to the work done in textile fibers since, I believe, 1840 might yield some useful information.

Mr. G. M. Kline.[3]—In line with the previous discussion I think that possibly we should attempt to divide the phenomena of flow of plastics under load into several categories as Mr. Delmonte has done in some of his papers. It would be of distinct interest if that type of information on recovery of plastics when the load is removed, particularly in the early stages, could be made available. This would be of value for many applications of cellulose acetate plastic in which relatively minor loads of short duration are involved.

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[1] Technical Director, Plastics Industries Technical Inst., Los Angeles, Calif.

[2] Physicist, E. I. du Pont de Nemours and Co., Arlington, N. J.

[3] Chief, Organic Plastics Section, National Bureau of Standards, Washington, D. C.

MR. W. N. FINDLEY[4] (*author's closure*).—In reply to the comments by Mr. Delmonte, the elapse of time between application of load and the first reading in a creep test was 15 to 20 sec. However, this interval of time is not as significant in the method of testing employed as Mr. Delmonte implies, because creep, as usually defined and as pointed out in the paper, is the total extension (measured in per cent) which has taken place up to a certain time. That is, creep includes both the initial elastic strain and the continuing deformation which takes place over a period of time. Thus the "elastic" part of the strain need not be determined. However it can always be obtained from a knowledge of the modulus of elasticity and the stress in the specimen.

Mr. Delmonte states that he observed creep at stresses as low as 100 psi. I do not wish to imply that no creep occurred at a stress of 500 psi. (the lowest stress used in the above tests) but that no creep was measurable with the instruments used. That is, the results of the above tests indicate that if creep occurred at a stress of 500 psi., it was less than 0.1 per cent in 5000 hr. It may be that the creep observed by Mr. Delmonte was due to the type of apparatus which he used.[5] In the cantilever beam specimen which Mr. Delmonte used there

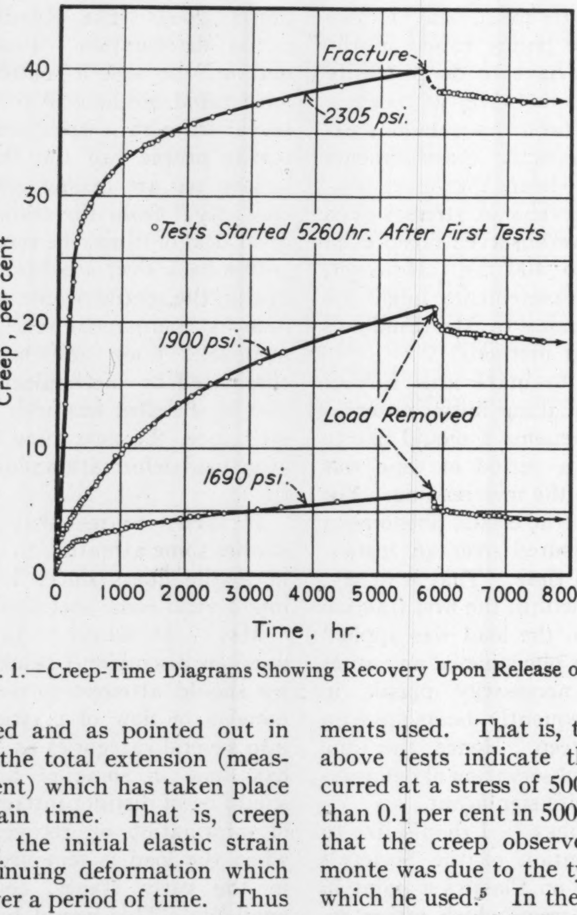

FIG. 1.—Creep-Time Diagrams Showing Recovery Upon Release of Load.

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[4] Associate in Theoretical and Applied Mechanics, College of Engineering, University of Illinois, Urbana, Ill.

[5] J. Delmonte and W. Dewar, "Factors Influencing Creep and Cold Flow of Plastics," ASTM BULLETIN, No. 112, October, 1941, p. 35.

are two points of stress concentration, one at the loading hook and the other at the clamp. His method of measurement would include deformations resulting from local high stress at both of these stress concentrations, so that the observed creep may have been the result of creep in the regions of high stress rather than creep at a stress of 100 psi.

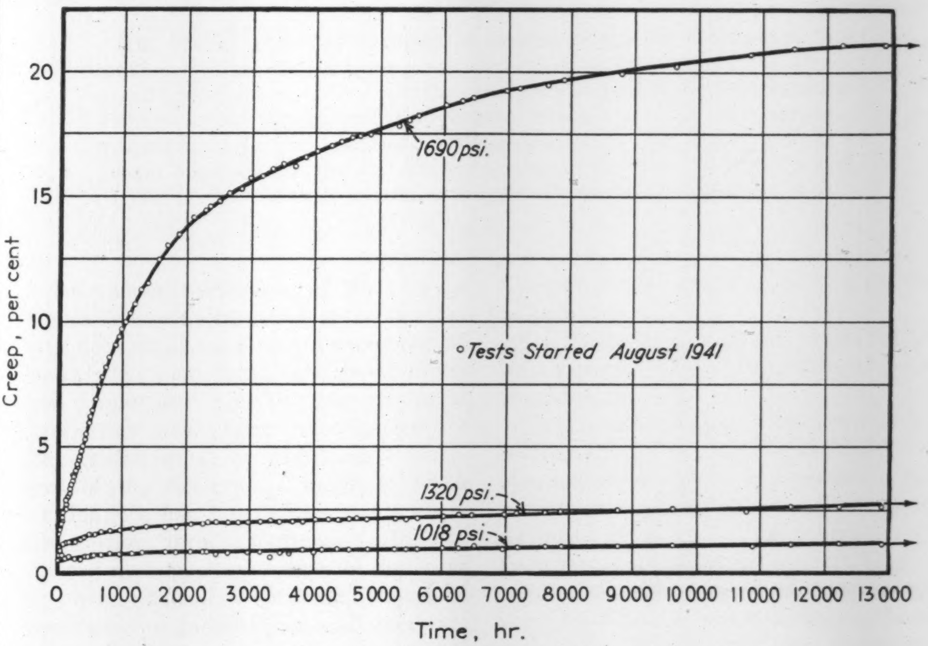

FIG. 2.—Creep-Time Diagram, 13,000 hr. (Compare Fig. 3).

As a result of comments by Mr. Kline and Mr. Cooper, data have been obtained on the recovery which occurs when the load is removed from a specimen after creep has taken place under a constant load. Tests at three different stresses are shown in the accompanying Fig. 1. The specimen which was stressed to 2305 psi. failed after about 5700 hr. The extensometer clips were, however, not disturbed by the fracture so that it was possible to record the relaxation after fracture. The specimens stressed to 1900 psi. and 1690 psi. were unloaded after about 5800 hr. These data are shown in the accompanying Fig. 1. The tests shown in this figure correspond to tests started 5260 hr. after the first tests (see Fig. 4). It was observed that the amount of recovery for tests at the three different stresses was about the same and that the rate of recovery (slope of curve) was also about the same even though the creep at the time the load was removed was as widely different as 40 per cent for the 2305 psi. stress and 6.5 per cent for the 1690 psi. stress. It, perhaps, is significant that the recovery after a relatively large amount of creep was of much smaller magnitude than the creep occuring over a like period of time. These recovery tests have continued for a period of about 1800 hr.

Since the paper was submitted, the original creep tests have been extended to a period of time of 13,000 hr., about 18 months. Data for the original group of specimens is shown in the accompanying Fig. 2. It was observed that the "first stage" of creep remained a straight line throughout the 18 months for the two lowest stresses shown, 1320 psi. and 1018 psi. The "second stage" of creep was found to be a gradual curve in the case of the 1690 psi. stress.

MAR RESISTANCE OF PLASTICS

By Ladislav Boor[1]

Synopsis

The special aspect of hardness of plastics tentatively designated "mar resistance" was studied by means of a test in which abrasive was allowed to fall against a test surface, and the resultant lowering of gloss was evaluated with an optical glossmeter. Comparative values are given for a variety of materials, plastic and nonplastic. Data obtained by this test on a series of plastic materials by three cooperating laboratories are given and a comparison is made of their relative rating by different test methods.

Increasing utilization of plastics as elements of structure and in the decorative field implies a constant expansion of knowledge concerning their basic physical properties. One of these groups of properties now under investigation by Subcommittee II on Hardness Properties of the Society's Committee D-20 on Plastics has been given the general descriptive term "hardness." In the field of metals, the property of hardness has been associated with the concept of resistance to indentation, and methods of measuring this property are in general use.

In the field of plastics, it was soon found that the term "hardness" was used to describe many aspects of the behavior of plastics, such as:

(*a*) Resistance to compression and the variation of this resistance with temperature.

(*b*) Resistance to indentation.

(*c*) Resistance to wear, as occurs when a shaft revolves in a plastic bearing.

(*d*) Resistance to scratching by abrasive particles as might occur in a plastic table top or tray.

(*e*) Resistance to optical degradation by air-borne abrasive particles, as in a plastic windshield.

That determinations of hardness under each of the above conditions would not rate a series of plastics in the same order was soon recognized. In Kline and Axilrod's investigation of properties of transparent plastics for aircraft (**1**),[2] a comparison was made of hardness measured by different indentation methods with that found by the Bierbaum microcharacter (**2**). Their conclusion at that time was:

> "It is therefore believed to be impractical to substitute the better known and more rapid indentation hardness measurement (Vickers) for scratch resistance data obtained with a sclerometer (Bierbaum)."

During the course of an investigation of scratch resistance of various thermosetting molding compounds and laminated decorative sheet materials, we attempted an evaluation of scratch hardness by means of the Bierbaum microcharacter. It was found that a rating on the basis of microhardness by the Bierbaum method did not agree too well with the results of service tests. This lack of correlation was partly due to the lack of precision inherent in the determination of microhardness of this type of material. The method requires measurement of a scratch made under standard conditions with an eyepiece scale in a microscope. The character of the furrows made in a brittle plastic surface layer makes it very difficult to judge the precise location of the edge of such a furrow to a high degree of certainty. For instance, in a group of samples, showing scratch widths of 29 to 36 μ, these values were accurate to about 4 μ, corresponding to a range of 7 to 12 points in microhardness values between 27.7 to 51.0. There is an additional error introduced by the elastic behavior of some of the plastics resulting in varying degrees of recovery at the edges of the furrow where the stresses are of a low order.

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[1] Physicist, American Cyanamid Co., Stamford Research Laboratories, Stamford, Conn.

[2] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 941.

A method of measuring abrasion resistance of paint films has been described by Schuh and Kern (3) and is now under consideration as a tentative standard by the Society's Committee D-1 on Paint, Varnish, Lacquer, and Related Products. In this method, a stream of abrasive is blown against a test surface and the amount of abrasive necessary to wear away a known thickness of paint film is determined. Some tests by this method on laminated plastics showed that the initial glossy surface of the material was removed in the first few seconds of the test, and further abrasion merely wore a depression into the body of the sheet. Weighing the amount of material removed in a short time was unsatisfactory because of the small weight losses involved.

It was later found that dropping a granular abrasive from a height of about 25 in. gave a milder abrasive action and that increasing amounts of abrasive produced abraded spots progressively duller in appearance.

This method is not new, having been in use by the author since about 1922 for evaluating abrasion resistance of cellulose nitrate film. Gardner (4) describes a similar method long used by his laboratory for testing paint and varnish films. More recently, the same principle has been used by Bruce Chalmers (5) for measuring the surface hardness of thin electroplated coatings, and its use on glass, plastics, and paint films was suggested.

In the earliest work mentioned, the abrasion resistance was expressed in terms of a set of prepared standards which were matched visually against the unknowns. Gardner (4) suggested the use of a glossmeter for rating gloss degradation of paint films and Chalmers (5) described an instrument for measuring specular gloss.

An experimental glossmeter had been built by the Stamford Laboratory for evaluation of paint films and coated papers which, with some minor changes, was adapted for measuring gloss of the abraded test areas on plastics. The problem was to obtain a sufficiently large and uniformly abraded spot upon which the incident light beam of the glossmeter could be focussed, and the spatial distribution of the reflected image analyzed. A large abraded area implied an abrasive stream of considerable cross-section—or a considerable quantity of abrasive if allowed to fall from a single opening—and the consequent interference of abrasive particles with each other. We found that by allowing the abrasive to fall from a hopper with small openings at the bottom arranged in annular fashion and rotating the hopper slowly, a uniformly abraded spot about $\frac{3}{4}$ in. in diameter could be produced, and the rate of abrasive feed would be low enough to minimize the interference of abrasive particles with each other.

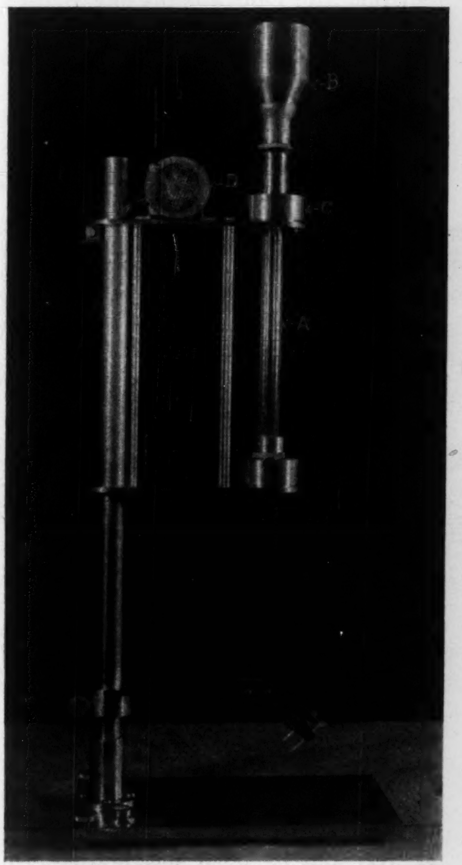

FIG. 1.—Photograph of Abrader.

The abrader as finally set up is shown in Fig. 1.[3] A glass tube *A*, 1 in. in outside diameter and $\frac{7}{8}$ in. in inside diameter is supported vertically. At the top is the rotating hopper *B*, driven by a small motor *D*. The hopper may be formed from thin sheet metal with smooth soldered seams. It has a double conical bottom with six holes 0.070 in. in diameter equally spaced in a circle $\frac{5}{8}$ in. in diameter at the intersection of the inner and outer cones, with one hole at the apex of the inner cone.

The ball bearing *C* is a standard item, $2\frac{1}{4}$ in. in outside diameter and $1\frac{1}{4}$ in. in inside diameter with sealed balls. The outside ring is rigidly clamped into position on a vertical stand. A brass or copper tube $1\frac{1}{4}$ in. in outside diameter and $1\frac{1}{8}$ in. in inside diameter and about $1\frac{1}{2}$ in. long is fitted tightly into the inner ring of the bearing. The glass tube extends upward into the metal tube and reaches just to the lower edge of the hopper. To the metal tube are soldered four supporting arms, inside of which the removable hopper fits. The upward projecting portion of the brass tube serves as a pulley which is driven by the small motor *D*[4], with built-in reducing gear at about 7 rpm.

The receptacle to receive the abrasive may be any metal container about 8 by 8 by 8 in. in size (a 5 gal. can, cut off to a height of 8 in. may be used) equipped with a supporting shelf at 45 deg. to the axis of the tube, spring clips to hold specimens under test, and slots at the ends to permit sliding sheet materials past the bottom of the abrasive tube. The height of drop, from the bottom of the hopper to the center of the test specimen at the axis of the tube, is 25 in. The abrasive used in our work was No. 80 carborundum which, with the construction shown, feeds from the hopper at about 200 to 250 g. per min. The assembly and alignment of the different parts should be made very carefully, and the axis of the tube should be exactly vertical as checked with a plumb line.

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[3] The Abrader, Fig. 1, and Glossmeter, Fig. 3, were designed and constructed under the direction of B. J. Dennison of the Pittsburgh Plate Glass Co., Creighton, Pa. Working drawings are to be available from A.S.T.M. headquarters.

[4] Speedway Manufacturing Co., 1834 S. Fifty-second Ave., Cicero, Ill. No. 952W. 11 rpm.

To make a test, the sample is clipped to the supporting shelf, the empty hopper started rotating, and the requisite amount of carborundum is poured into the hopper. Suggested amounts of abrasive for each separate test are 200, 400, 800, 1200, and 1600 g. When a given amount of abrasive has dropped through the sample is shifted and another spot is made with the next succeeding amount. There are finally five abraded spots each produced by the respective amount of abrasive. These areas may have particles of abrasive clinging to or embedded in the surface. Wiping or brushing of the test area has been found to give irregular and nonreproducible results. A mild air blast has been satisfactorily used to clean off excess abrasive and is recommended.

The abraded spots will appear to the eye as dulled areas about $\frac{3}{4}$ in. in diameter, of uniform dullness across the entire test area. Misalignment of parts or improper feeding of abrasive will result in areas of nonuniform appearance, such as edges duller than the center, or *vice versa*. Adjustments should be made until the test spot appears visually uniform.

It has been observed that continued use of the same batch of carborundum causes an apparent improvement of mar resistance of a given material, probably because of breaking up of abrasive particles or by dulling their sharp cutting edges. We have tentatively set a limit of 50 runs for a given lot of abrasive after which it is discarded. It is also advisable to keep this test abrasive well screened, passing a No. 60 sieve to take out extraneous matter such as lint, hairs, wood splinters, etc., and retained on a No. 120 sieve to remove fine broken-down abrasive particles and dust.

The particular aspect of hardness of which this test is a measure has been given the tentative designation "mar resistance."

Depending on the type of plastic under consideration, various methods of evaluating the optical degradation of the test areas may be used. For transparent plastics some type of haze meter, such as the Kline Bowen Instrument (1), the American Optical Co.[5], the Barnes Sphere turbidimeter[6], or any of the instruments described in the Proposed Tentative Method of Test for Measuring Photoelectrically the Haze of Transparent Plastics[7] may be used, and the results expressed in percentage of haze, or percentage transmission against quantity of abrasive. Since most of our work has been concerned with opaque plastics, the simplified glossmeter previously referred to was used..

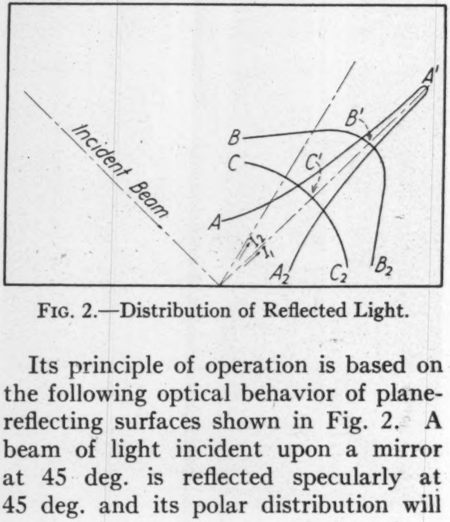

FIG. 2.—Distribution of Reflected Light.

Its principle of operation is based on the following optical behavior of plane-reflecting surfaces shown in Fig. 2. A beam of light incident upon a mirror at 45 deg. is reflected specularly at 45 deg. and its polar distribution will have the shape AA_1A_2. If the mirror surface is disturbed by abrasion, progressively more light will be scattered on either side of the specularly reflected beam and its distribution will assume the shape BB_1B_2 approaching the uniform distribution CC_1C_2 characteristic of a nearly perfect diffusing surface such as magnesium carbonate.

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[5] H. R. Moulton, Proposed Method of Haze Measurement, Report to Subcommittee IV on Optical Properties of A.S.T.M. Committee D-20 on Plastics.

[6] Barnes turbidimeter now under development.

[7] Report of Committee D-20 on Plastics, see p. 453.

If we place a lens in the path of the reflected beam and focus the image of the illuminated spot on a photocell and measure its intensity at the specular angle I_1 and again along the angle I_2 at 15 deg. away from the specular, their ratio is a measure of the peakedness of the light distribution curve, or a measure of the gloss. The angle of 15 deg. off specular was chosen, because with the particular instrument described, the specular beam from a mirror surface just misses the opening of the receptor lens. Figure 3 is a photograph of the instrument.[3]

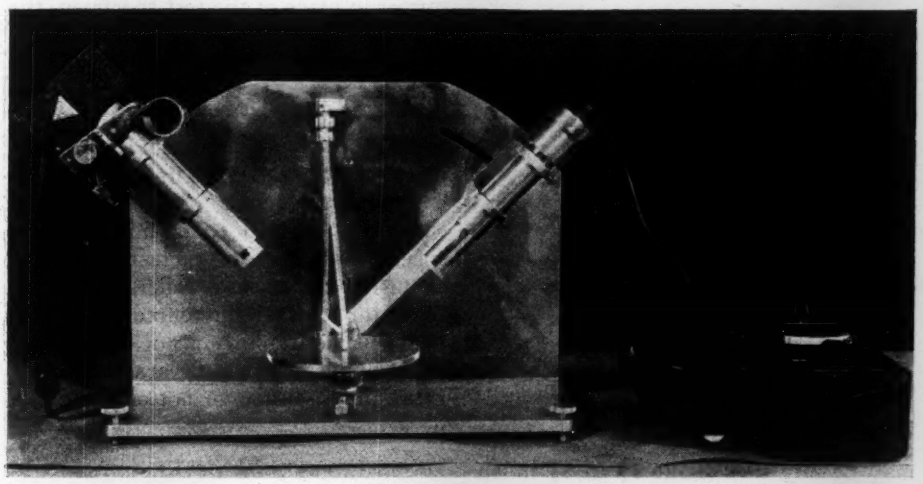

FIG. 3.—Glossmeter.

To reduce the complication caused by variable photocell readings from surfaces of differing original reflectivity and to minimize the effect of voltage variation on the intensity of the incident beam, only the ratio of light intensities reflected at the two observation angles is used in the gloss formula.

If I_1 is the photocell reading at the specular angle and I_2 is the reading at the 30 deg. angle (15 deg. off specular), then

$\frac{I_1}{I_2}$ is a measure of the sharpness of the peak of light distribution.

To convert this into convenient numerical terms,

$$\text{Gloss, per cent} = 100\,\frac{I_1 - I_2}{I_1}$$

Accordingly, a zero reading at the off-specular angle gives 100 per cent gloss and equal readings at specular and off-specular angles correspond to 0 per cent gloss.

In comparing abrasion characteristics of a series of materials which do not have the same original gloss, the initial gloss value of the unabraded surface is equated to 100 per cent and the progressive degradation is expressed in per cent.

Typical data are shown in Table I.

The glossmeter may be assembled from the following parts:

The light source is a Model "M"

S.V.E. Projector.[8] An extension tube is made to permit the lens to form an image at a short distance. The size of the test spot is determined by several circular holes ranging from $\frac{1}{8}$ to $\frac{3}{8}$ in. in diameter, punched into a piece of blackened 35-mm. film, and loaded into the film carrier. To regulate the quantity of light, independent of the size of the test spot, a diaphragm may be added to the front of the projector lens. This may be a microscope condenser iris diaphragm with a $1\frac{1}{4}$-in. opening fitted over the projector lens, or a series of caps over the front of the lens with openings of varying diameters in steps of $\sqrt{2}$.

TABLE I.

Abrasive, g.	Photocell Readings		Gloss, per cent	Percentage of Original
	I_1	I_2		
0	93.5	2.0	97.9	100
200	60.0	4.0	93.3	95.4
400	43.5	5.0	88.5	90.4
800	33.0	6.0	81.8	83.5
1200	25.0	6.0	76.0	77.6
1600	19.0	6.0	68.4	70.0

I_1 = Reading at specular (45 deg.) angle.
I_2 = Reading at off specular (30 deg.) angle.
Gloss, per cent $= 100 \frac{I_1 - I_2}{I_1}$.

The pickup for the reflected beam may be any lens about $1\frac{1}{4}$ to $1\frac{1}{2}$ in. in diameter of about 4 to 5-in. focal length (a duplicate of the projector lens may be used) which picks up the reflected beam and converges it sufficiently to impinge on the sensitive area of the photocell. The photocell may be of any type having a linear current-intensity relation.[9] The projector may be permanently mounted with its optical axis at 45 deg. to the plane of the specimen with a distance of about 12 in. from the perforated film to the center of the test surface. The receptor system may be fastened to a hinged arm movable about a point located in the plane of the test surface at the center of the illuminated test spot, with freedom of motion from a position at 45 deg. to the plane of the specimen, to the off-specular position at 60 deg. to the plane of the specimen.

When properly assembled, and with a mirror in place of the test specimen, the circular reflected image of the incident light beam should be seen entering the receptor lens at the specular 45-deg. angle. With the arm tilted upward 15 deg., this specular beam should just miss the receptor lens in its new position. The active surface of the photocell should be masked to permit only the specularly reflected image of the test spot to strike the cell. A voltage regulator on the light source in the projector is desirable but not essential if the voltage fluctuations are not of too great an amplitude, or not of very short period.

EXPRESSION OF RESULTS

Having obtained the values of percentage gloss after varying amounts of abrasive action, these may be plotted against each other giving a characteristic curve. Such curves for different materials often are found to change slope irregularly, depending on the degree of "case hardening" of the surface layer; they may cross each other, and the rating of a series at a given amount of abrasive may not be representative of their relation at another amount. One method of arriving at an over-all performance index is to average the percentage of original gloss at the various amounts of abrasive, which would ap-

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[8] Society for Visual Education, 100 E. Ohio St., Chicago, Ill.

[9] Any barrier layer cell having an active surface about 1 in. in diameter may be used, such as No. 732 "Electrocell," Photovolt Corp., 95 Madison Ave. General Electric and Westinghouse make similar cells. The galvanometer may be General Electric No. 32C 249 G25 with built-in shunts covering ranges of 10–5–2–1 or a similar instrument having an internal resistance of 300 ohms or less. Other units which include both photocell and meter are: Photrix Universal Photometer Model A., Photovolt Corp., 95 Madison Ave., New York, N. Y.; Weston Model 603 Illumination Meter with single No. 594 cell, Weston Electrical Instrument Corp., Newark, N. J.

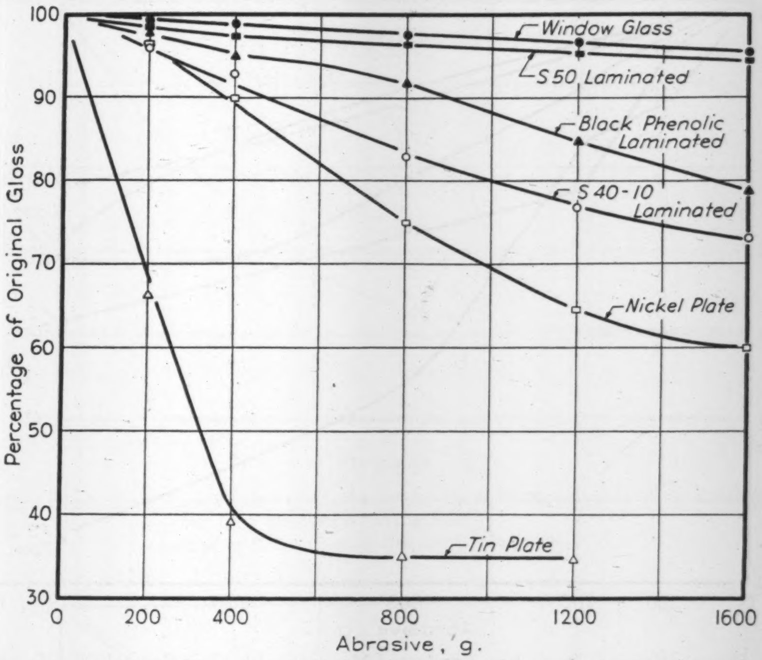

FIG. 4.—Shows Tests on Window Glass, Melamine-Urea Resin Surface Laminated (S50), Black Phenolic Paper Base Laminated, Plasticized Melamine-Urea Laminated (S40-10), Nickel Plate, and Tin Plate.

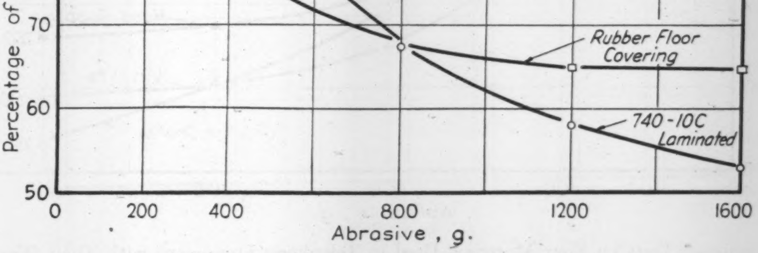

FIG. 5.—Shows Black Ferrotype Plate, Cellulose Acetate Sheet, a Plasticized Melamine-Urea Laminated (740-10C), and a Rubber Floor-Covering Compound.

A test on rubber in the form of tire tread stock gave a line which varied but slightly from 100 per cent and on which the abraded spots were hardly distinguishable.

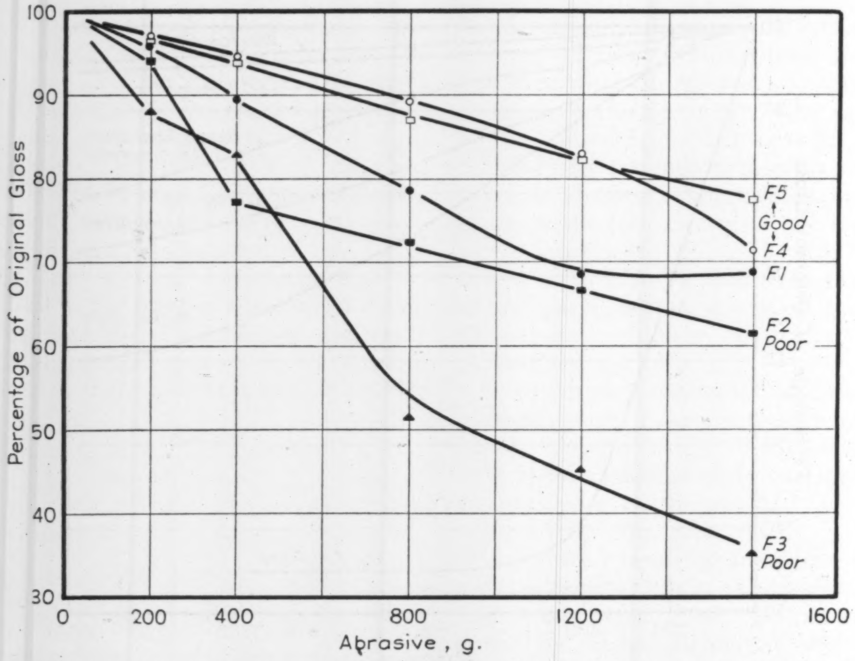

FIG. 6.—Shows a Group of Paper-Base Laminated Materials with Their General Classification Based on Service Tests.

Samples from Formica Insulation Co., Cincinnati, Ohio.

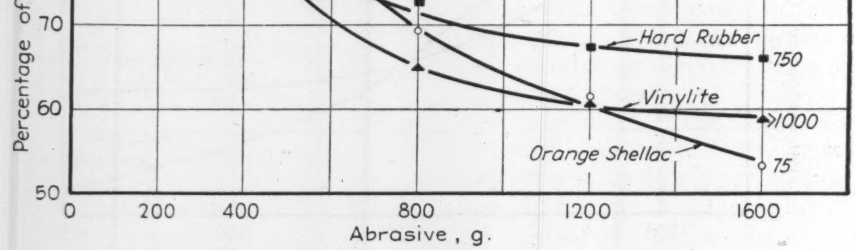

FIG. 7.—Shows Tests on Four Materials Used in Telephone Equipment with Their Wear Indices Based on Service Tests.

The correlation in this group is poor.

Samples submitted by Robert Burns, Bell Telephone Laboratories.

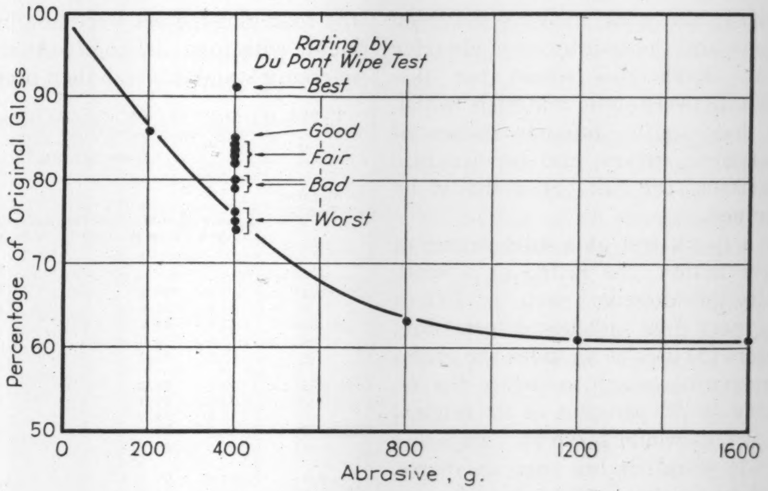

FIG. 8.—Represents Tests on a Variety of Methacrylate Sheet Materials with Their Ratings on the Basis of a "Wipe Test".

Submitted by E. I. du Pont de Nemours Co., Arlington, N. J.

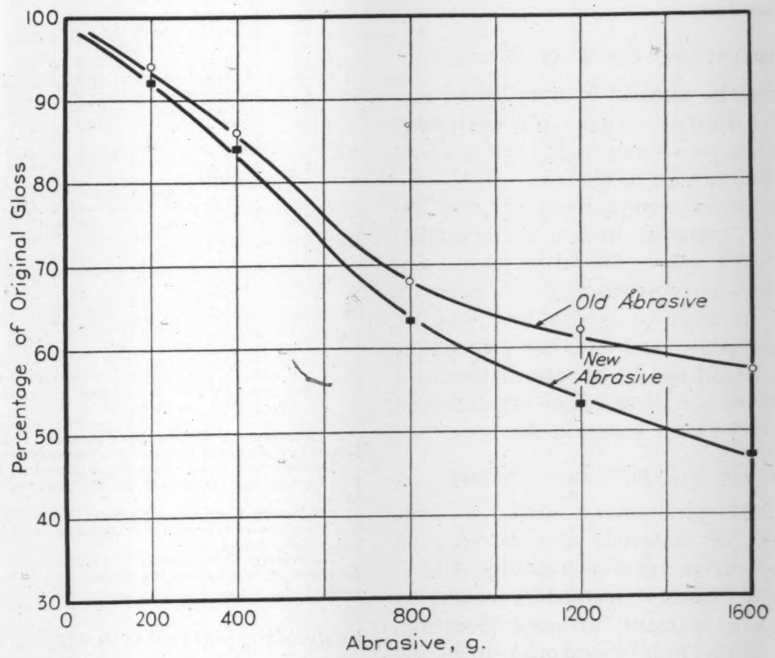

FIG. 9.—Shows the Effect of Using Fresh and Old Abrasive in Testing the Same Material. The "old" abrasive had been used for about 400 runs.

proximate the area included between the curve and the coordinate of abrasive amount. Even this would not distinguish between two materials which might have quite different shapes of characteristic curves, and for accurate comparisons, the full curve should be determined.

For a quick test at a mild degree of abrasive action, the rating at a small quantity of abrasive, such as 200 or 400 g., may give sufficient information. Chalmers (5) uses as an index the grams of abrasive necessary to reduce the reflectivity to 50 per cent of its original value. This would serve for comparing materials of rather low mar resistance, but might entail considerable time to obtain a sufficient number of points which would be extrapolated to 50 per cent of original gloss, on materials such as glass.

LIMITATIONS OF TEST METHOD

Since the method is intended to rate relative decrease in gloss, obviously only materials possessing highly specular reflectance can be measured.

For strict comparisons of mar resistance, materials having nearly similar reflectance values should be used. For instance, a comparison of two types of resin coatings in which one was an opaque white and another an opaque black would not be as informative as a test made on these resins compounded to give the same general color.

Evaluation of Typical Test Materials:

In the preliminary work, a large number of materials was tested, and typical curves are shown in Figs. 4 to 9.

These results seemed encouraging enough to warrant further exploration. Accordingly, four laboratories (including our own) agreed to carry out a series of cooperative tests. After one unsuccessful attempt it was apparent that the four glossmeters were not in agreement with one another. A series of reference standards was then prepared,[10] by grinding plate glass to varying degrees of surface roughness, calibrating them on one instrument, and distributing

TABLE II.—COMPARISON OF CALIBRATION.

Standard Sample	Gloss, per cent[a]				
	Laboratory A	Laboratory B	Laboratory C	Laboratory D	Difference from Laboratory A
No. 1 *a*......	99.3	99.4	....	....	+0.1
b......	80.8	79.1	....	....	−1.7
c......	83.0	81.4	....	....	−1.6
d......	77.0	80.0	....	....	+3.0
e......	45.0	47.0	....	....	+2.0
No. 3 *a*......	99.3	99.4	....	....	+0.1
b......	90.5	90.0	....	....	−0.5
c......	84.8	85.0	....	....	+0.2
d......	79.5	78.7	....	....	−0.8
e......	46.8	46.8	....	....	0
No. 4 *a*......	99.3	99.3	....	....	0
b......	72.0	73.6	....	....	+1.6
c......	81.8	80.6	....	....	−1.2
d......	76.0	74.5	....	....	−1.5
e......	46.3	46.7	....	....	+0.4
No. 6 *a*......	99.3	99.3	....	....	0
b......	89.0	90.0	....	....	+1.0
c......	76.0	79.3	....	....	+3.3
d......	73.4	73.5	....	....	+0.1
e......	45.5	46.7	....	....	+1.2
No. 5 *a*......	99.3	....	99.8	....	+0.5
b......	89.5	....	88.0	....	−0.5
c......	82.0	....	79.9	....	−2.1
d......	73.8	....	73.6	....	−0.2
e......	45.0	....	47.1	....	+2.1
No. 2 *a*......	99.3	....	....	99.5	+0.2
b......	91.0	....	....	93.3	+2.3
c......	86.0	....	....	88.6	+2.6
d......	78.8	....	....	79.5	+0.7
e......	46.7	....	....	49.2	+2.5
No. 7 *a*......	99.3	....	....	....	....
b......	90.5	....	....	....	....
c......	82.0	....	....	....	....
d......	75.8	....	....	....	....
e......	47.4	....	....	....	....
No. 8 *a*......	99.3	....	....	....	....
b......	87.0	....	....	....	....
c......	79.8	....	....	....	....
d......	72.5	....	....	....	....
e......	46.7	....	....	....	....

[a] Laboratory A = American Cyanamid Co., Stamford Laboratory; Original Laboratory Instruments.

Laboratory B = Pittsburg Plate Glass Co., Creighton, Pa.; Abrader and Glossmeter designed and built by themselves.

Laboratory C = Brooklyn Navy Yard, Brooklyn, N. Y.; Abrader and Glossmeter built according to Pittsburg Plate Glass design.

Laboratory D = Brooklyn Polytechnical Institute, Brooklyn, N. Y.; Laboratory instruments built by themselves.

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[10] These were prepared through the kindness of B. J Dennison of the Pittsburgh Plate Glass Co

them among the four laboratories. The proper adjustments were then made on each of the instruments, and the final comparison of calibration is given in Table II. It is our opinion that this agreement between instruments of different makes using different photocells and electrical measuring equipment is as good as can be expected. Higher degrees of precision would result from the construction of all instruments by the same company, but this must be left for the future.

A series of plastic sheet materials was then distributed among three of the laboratories, each equipped with its own abrader and glossmeter. The comparisons were made on the basis of the following amounts of abrasive: 400, 800, 1200, and 1600 g. Table III shows the values obtained with the average of

TABLE III.—VALUES OBTAINED WITH THE AVERAGE OF RESIDUAL GLOSS.

Laboratory	Material[a]	Percentage of Original Gloss				
		400 g. Abrasive	800 g. Abrasive	1200 g. Abrasive	1600 g. Abrasive	Avg.
B	Formica No. 1	90.3	80.5	72.9	65.0	77.2
C		87.8	80.0	70.8	66.0	76.2
A		89.0	79.5	73.1	67.3	77.2
B	Formica No. 2	69.0	49.9	36.8	26.8	45.6
C		75.6	55.7	42.2	41.2	53.7
A		76.1	62.5	51.8	45.4	59.0
B	Formica No. 3	83.7	64.7	51.1	43.8	60.8
C		80.2	62.3	47.8	34.5	56.2
A		78.8	62.4	53.1	50.6	61.2
B	Polystyrene	45.9	27.8	29.1	24.5	31.8
C		43.1	22.1	20.5	17.9	25.9
A		50.8	39.1	33.9	33.9	39.4
B	Vinylite	76.6	59.2	53.5	48.5	59.5
C		75.4	58.7	45.2	43.4	55.7
A		74.1	65.4	62.0	59.1	65.2
B	Cellulose acetate	84.6	74.3	63.9	60.2	70.8
C		81.7	68.7	59.4	51.2	65.3
A		82.7	72.6	67.4	63.1	71.5
B	Methacrylate	81.4	61.1	47.6	46.7	59.2
C		77.4	58.5	39.6	27.2	50.7
A		74.1	60.7	56.1	53.5	61.1

[a] Formica No. 1—Laminated sheet material, walnut color, urea-melamine resin surface.

Formica No. 2—Laminated sheet material, light green color, urea-thiourea resin surface.

Formica No. 3—Laminated sheet material, mahogany color, phenolformaldehyde resin surface.

Formica Nos. 1, 2, and 3 obtained from J. D. Pitzer, Formica Insulation Co., Cincinnati, Ohio.

Polystyrene—Transparent amber injection molded sheet No. 141-A7014-A. Obtained from H. K. Nason, Monsanto Chemical Co., Springfield, Mass.

Vinylite—Opaque dark blue sheet VS 3300. Obtained from M. C. Reed, Vinylite Division, National Carbon Co., Cleveland, Ohio.

Cellulose Acetate—colorless transparent sheet. Obtained from E. Schweizer, The Celluloid Co., Newark, N. J.

Methacrylate—Colorless transparent sheet. Obtained from the E. I. du Pont de Nemours Co., Arlington, N.

TABLE V.

	Mar Resistance				Scratch Hardness			
	Average	Rating	400 g.	Rating	Bierbaum[a]	Rating	Olsen[b]	Rating
Formica No. 1....	76.9	1	89.0	1	30.9	2	2.27	2
Cellulose acetate..	69.2	2	82.7	2	12.2	6	0.89	6
Vinylite....	60.1	3	75.4	5	12.2	7	0.70	7
Formica No. 3....	59.4	4	80.9	3	21.4	3	1.88	3
Methacrylate...	57.0	5	77.6	4	21.4	4	1.88	4
Formica No. 2....	52.8	6	73.6	6	47.6	1	2.81	1
Polystyrene.....	32.7	7	46.6	7	15.8	5	1.35	5

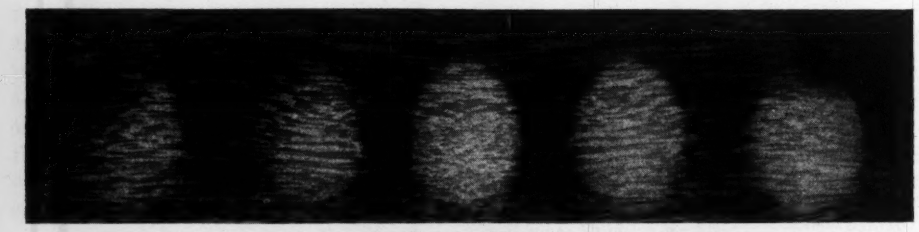
[a] Bierbaum Scratch Hardness tests made with micro character (**2**).

[b] Olsen Scratch Test made on an instrument now under development. Data from a report to Subcommittee II of Committee D-20 by B. L. Lewis, September 26, 1941.

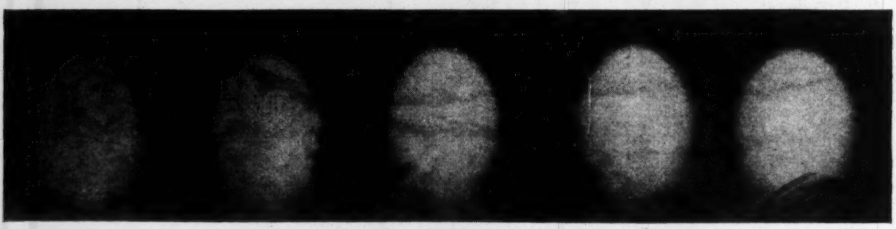
TABLE IV.

	Laboratory A				Laboratory B				Laboratory C			
	Average	Rating	400 g.	Rating	Average	Rating	400 g.	Rating	Average	Rating	400 g.	Rating
Formica No. 1..................	77.2	1	89.0	1	77.2	1	90.3	1	76.2	1	87.8	1
Cellulose acetate..................	71.5	2	82.7	2	70.8	2	84.6	2	65.3	2	81.7	2
Vinylite..................	65.2	3	74.1	5	59.5	4	76.6	5	55.7	4	75.4	6
Formica No. 3..................	61.2	4	78.8	3	60.8	3	83.7	3	56.2	3	80.2	3
Methacrylate..................	61.1	5	74.1	6	59.2	5	81.4	4	50.7	6	77.4	4
Formica No. 2..................	59.0	6	76.1	4	45.6	6	69.0	6	53.7	5	75.6	5
Polystyrene..................	39.4	7	50.8	7	31.8	7	45.9	7	25.9	7	43.1	7

residual gloss at the four amounts of abrasive.

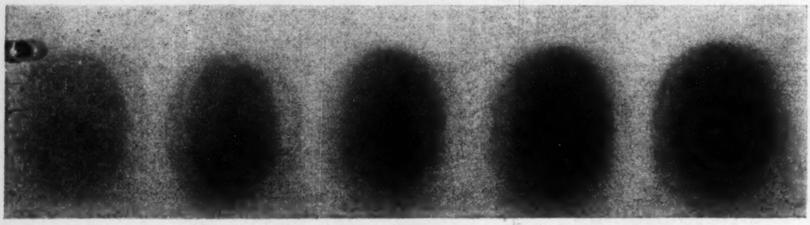


Formica No. 1

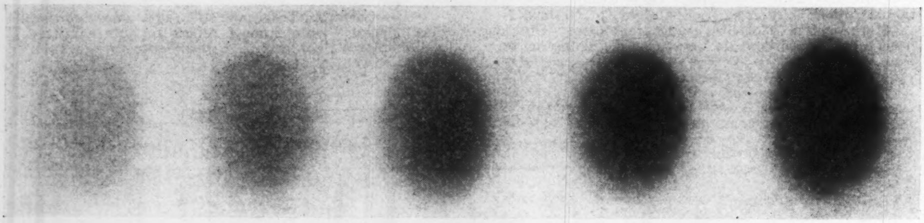


Formica No. 3

FIG. 10.—Abraded Test Areas on Typical Laminated Plastics.



Polystyrene



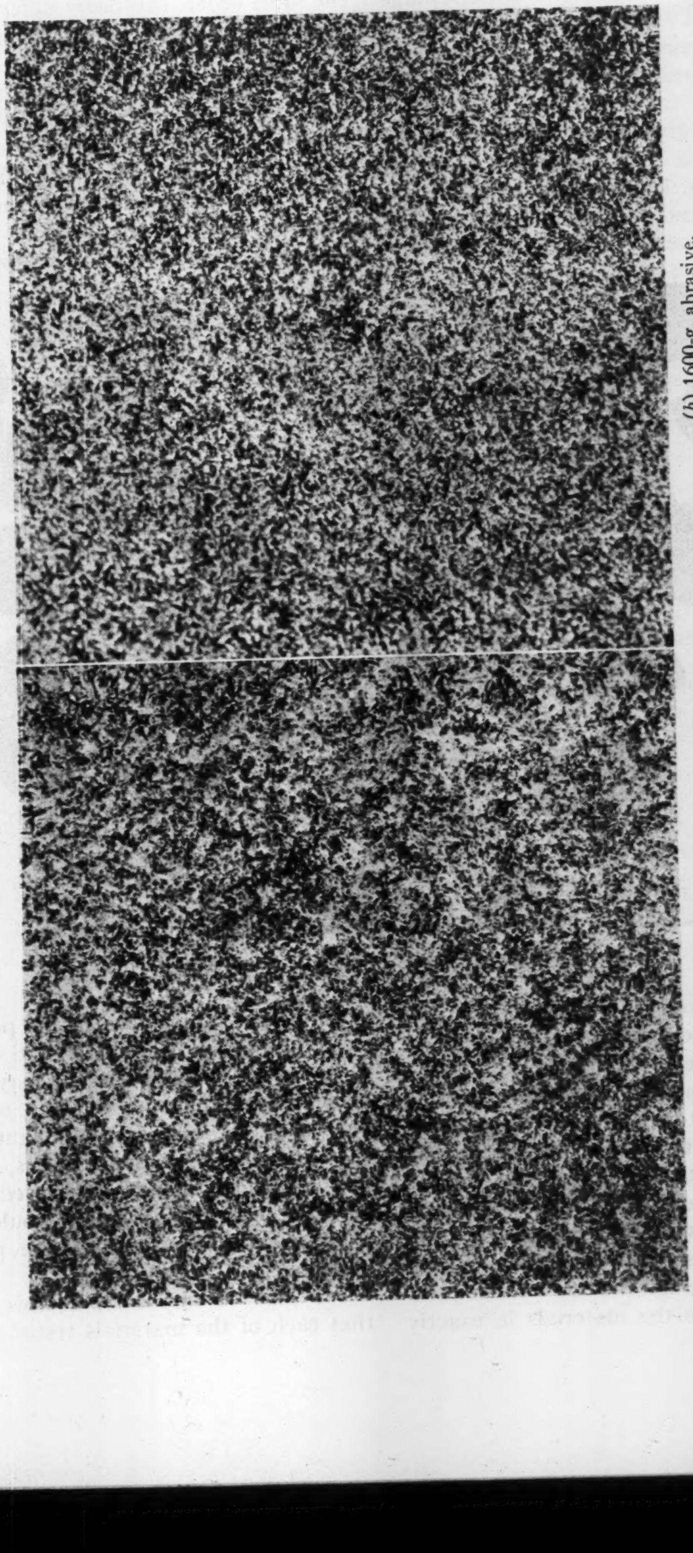
Methyl methacrylate

FIG. 11.—Abraded Test Areas on Typical Transparent Plastics.

INTERPRETATION OF DATA

These data may be interpreted in two general ways. Recognizing the fact that all plastic materials are not uniformly hard as the surface is penetrated, we might compare them on the basis of:

(*a*) The average area (up to 1600 g.



(a) 200-g. abrasive. (b) 1600-g. abrasive.

FIG. 12.—Abraded Test Areas of Methacrylate Sheet (X 200). Transmitted light.

abrasion) under the curve of percentage gloss plotted against amount of abrasive.

(*b*) The relative percentage gloss at a constant small amount of abrasive (400 g.).

Table IV gives these values.

It appears that the two materials Formica No. 1 and cellulose acetate are rated first and second, and polystyrene is rated seventh by all laboratories. The other four materials are closely grouped between these two extremes, by all laboratories.

Table V, based on the averages of all three laboratories on each material, gives the relative abrasion resistance of the group, rated at average for the full 1600-g. test and at 400 g. of abrasive. For comparison the ratings by two methods of scratch hardness are included. Although the two scratch hardness tests rate the materials in exactly the same order, this order differs widely from the rating by the mar resistance test.

It can be seen that continued abrasion will reduce the gloss of any plastic until its surface approaches a nearly constant matte condition; hence small differences in the rate of degradation at the beginning of the test are masked by the approach to a constant value with large amounts of abrasion. It is possible that the relative gloss at a mild degree of abrasion, such as 400 g., may be a sufficiently significant measure of mar resistance. A test at one quantity is shorter than the complete series, where five different amounts are used, and several check runs at 400 g. should give a representative value for the plastic under test.

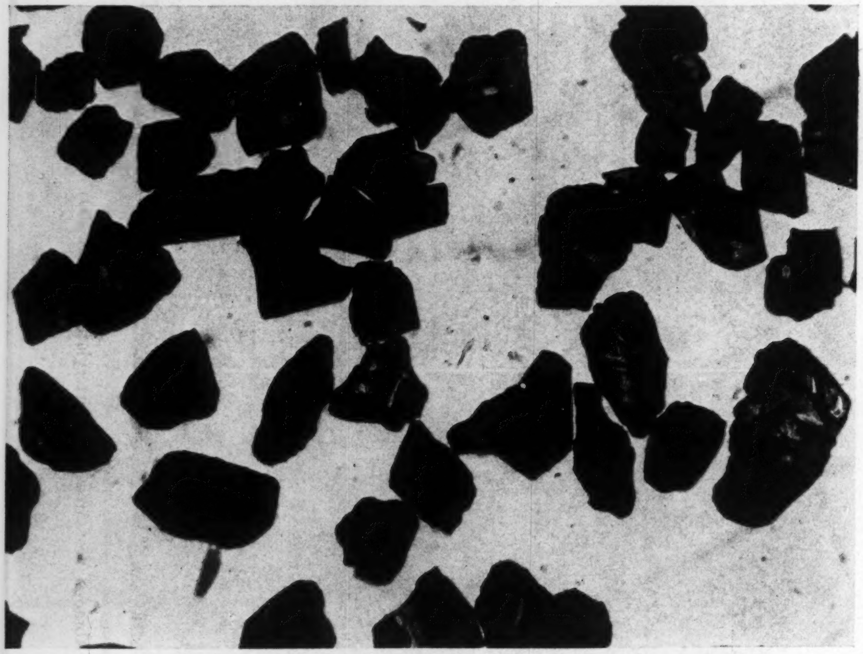


FIG. 13.—Photomicrograph of No. 80 Carborundum (X 50).

We wish to emphasize at this point that each of the materials tested represents only one of a wide variety of formulations and range of properties which can be imparted to any one of the group; hence the ratings of scratch and mar resistance as shown cannot pretend to represent a final and absolute rating of the materials listed.

Figures 10 and 11 show the appearance of typical test specimen after abrasion. Figure 12 shows photomicrographs of abraded areas of methacrylate sheet. Figure 13 shows photomicrograph of No. 80 carborundum.

Acknowledgments:

We wish to express our indebtedness to R. B. Barnes, C. R. Stock, and J. R. Schneider of the Stamford Laboratories staff for their assistance in the preparation of this paper; to B. J. Dennison of the Pittsburg Plate Glass Co. for his share in the design of the Abrader and Glossmeter illustrated, and his help in the preparation of gloss standards; to the laboratories whose test work provided the basis for most of our data; and lastly to the members of Committee D-20 who furnished the materials tested.

REFERENCES

(1) G. M. Kline and B. M. Axilrod, "Study of Transparent Plastics for Use on Aircraft," *Journal of Research*, Nat. Bureau Standards, Vol. 19, October, 1937, pp. 367–400 (*RP 1031*).

(2) C. H. Bierbaum, "A Study of Bearing Metals," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 69, pp. 972–989 (1923).

(3) A. E. Schuh and E. W. Kern, "Measurement of Abrasion Resistance," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 3, pp. 72–76 (1931).

(4) Henry A. Gardner, "Physical and Chemical Examination of Paints, Varnishes, Lacquers and Colors," Ninth Edition, p. 128 (1939).

(5) Bruce Chalmers, "Surface Hardness of Metals," *Journal* Inst. of Metals (British), Vol. 67, pp. 295–314 (1941).

DISCUSSION

Mr. R. S. Hunter.[1]—I have two observations to make regarding the method of measuring gloss.

(1) The optical system, which has been designed to deal with small test areas, is such that it will be necessary to make careful comparisons of one instrument with another to get results that are comparable from one laboratory to another.

(2) I wish to comment favorably on Mr. Boor's method of interpreting the gloss data; I think it well to subtract the reading the instrument gives at the off gloss angle, that is, 60 deg., as Mr. Boor calls it, from the reading at the specular angle, that is, 45 deg.

Mr. H. R. Moulton.[2]—At the time this problem of mar resistance and, in general, the abrasion resistance of plastics, first came up, the discussion centered around the general term "resistance to scratching." It is interesting to see that in this paper and in the discussion of the work which has been done it has been referred to as "mar resistance," and especially classified as "mar resistance due to air-borne abrasive."

There is, however, another field of mar resistance that I feel should be investigated, and that is the general abrasion resistance. A great deal of work has been done—I know a great deal of work has been done at the National Bureau of Standards by Mr. Kline and some has been done by Mr. Dennison of the Pittsburgh Plate Glass Co.—and in the discussions which we have had in the subcommittee on this particular subject there have been a number of suggestions offered.

The injury to a surface which results from wiping that surface is one far more difficult to evaluate, I think, than the injury to the surface that results from air-borne abrasive, and it seems to me that work should be done along that line. I have no suggestions to offer. I have tried perhaps half a dozen different methods to evaluate the wiping durability of abrasive surfaces and I have found none that I would depend upon 100 per cent. However, after studying the results and talking with the people who have run the tests with this method I would place considerable confidence in evaluating a series of plastics of a variety of types in the proper order of resistance to mar resistance by air-borne abrasive using this method.

Mr. Hunter.—Regarding Mr. Moulton's comment, the Porcelain Enamel Institute has done a thorough job of preparing methods to test the resistance of porcelain enamel surfaces to what they call abrasion. They are interested in the wiping resistance of porcelain enamels to cleaning with so-called cleaning compounds which "don't scratch," and they have a test which is now standard with them. The Porcelain Enamel Institute test might be a good place to start.

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[1] Assistant Physicist, National Bureau of Standards, Washington, D. C.

[2] Assistant Director of Research, American Optical Co., Southbridge, Mass.

MR. W. H. GARDNER.[3]—The characteristics of plastics and that of vitreous enamel are quite different. The aspects of the vitreous enamel tests were considered in developing this particular test. With glass you do not encounter the elastic properties, and the problem of rubbing is quite different than what we are attempting to measure here.

MR. H. E. SMITH.[4]—It may be of interest to mention an abrasion test on an entirely different material. Some years ago we were desirous of testing the abrasion resistance of locomotive bell cords. We tried rubbing the cord over all sorts of things to get something that would give uniform and instructive results. We finally settled on a glass tube of such character that it was fairly uniform.

The tube was supported securely in a horizontal position. A suitable weight was attached to the lower end of the test specimen of cord. The cord was passed over the tube and out horizontally behind it, the weight hanging on the cord and vertically below the tube. The upper end of the cord was attached to a suitable mechanical device by which it could be reciprocated horizontally over about 5-in. stroke. Locomotive water level gage tubes, which are of tough, uniform glass were satisfactory.

The method was rather crude but the principle involved might serve in the present case.

MR. LADISLAV BOOR[5] (*author's closure, by letter*).—It is recognized that the method described measures only one aspect of the resistance of plastics to surface scratching. In cases where the criteria of acceptability concern very small departures from a specularly reflecting surface, the method may have a field of usefulness. When the amount of "wear" beyond this point is desired, other procedures may prove to be more indicative.

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[3] Chief, Chemical Section, Specifications Branch, Conservation Division, War Production Board, Washington, D. C.

[4] Materials Engineer, White Plains, N. Y.

[5] Physicist, American Cyanamid Co., Stamford Research Laboratories, Stamford, Conn.

THE INFLUENCE OF TEMPERATURE ON THE MECHANICAL PROPERTIES OF MOLDED PHENOLIC MATERIALS

By T. S. Carswell,[1] D. Telfair,[1] and R. U. Haslanger[1]

Synopsis

Data showing the variation of the impact, flexural, and tensile strengths with temperature are given for molded phenolic compositions containing wood flour, asbestos, macerated fabric, and cord fillers and for a pure resin composition. Impact values between −80 C. and 250 C. (−112 F. and 480 F.), flexural values between −80 C. and 180 C. (−112 F. and 355 F.) and tensile values between −80 C. and 230 C. (−112 F. and 445 F.) are presented. The practical importance of these data are discussed.

A knowledge of the effect of temperature on the mechanical properties of molded compositions is becoming increasingly important with the growing demand for plastics in structural uses. Many molded parts for military purposes (**1, 2, 20**)[2] are going into applications which require that they be serviceable over the temperature range from −51 C. to 70 C. (−60 F. to 160 F.) As a result, specifications are being written by the Army and Navy to cover the mechanical properties of plastics at these temperatures. Requests have recently been received from various divisions of both Services for data on this subject. Numerous aircraft manufacturers using plastics in structural applications, as well as the National Advisory Committee for Aeronautics, have expressed a vital need for figures showing the change of mechanical strengths with temperature.

Relatively little significant information on the mechanical properties of plastic materials intended for structural uses has been available in the literature until recently. A publication in 1939 by Jacobi and Thum (**3**) describes in great detail the physical properties of phenolic compositions at room temperature. Additional information is given in articles by de Bruyne (**4**), Genung (**5**), Melville (**6**), Haut (**7**), and others (**8, 9, 10**). In general, these workers have failed to emphasize the importance of changes in temperature on the physical properties under consideration.

Within the last year Delmonte (**11**) has reported on the shear strength of molded phenolic materials over a limited temperature range. The variation of flexural strength and the deflection at failure for a number of compression molded plastics at temperatures from −70 C. to 200 C. (−94 F. to 390 F.) is described by Nitsche and Salewski (**12**), while a determination of static tensile, compressive, stiffness, and bending properties, and fatigue strengths of three laminated phenolic materials at −39 C., −18 C., and 25 C. (−38 F., 0 F. and 77 F.) are included in *Air Corps Technical Report No. 4648* (13). Kistler (21) and Kozlov (22) have also reported on the effect of temperature on the mechanical properties of plastics.

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[1] Research Director, Research Physicist and Research Chemist, respectively, Plastics Division, Monsanto Chemical Co., Springfield, Mass.

[2] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 953.

Almost no previous work has been reported on the effect of temperature on the impact strength or of elevated temperature or the tensile strength of molded phenolic compositions. Research work was undertaken to study the change of impact, tensile, and flexural strengths over the temperature range from −80 C. to 250 C. (−112 F. to 480 F.). Molded compositions employing wood flour, asbestos, macerated fabric, and cord fillers as well as a pure resin composition were evaluated. Creep properties, shear, and compressive strengths for these materials are also being studied over the same temperature range and will be reported at a later date.

The data reported here are short-time temperature effects. The effect of extended conditioning at temperatures other than room temperature, 25 C. (77 F.), has been touched upon briefly by Place (14) who studied phenolic laminated materials over a limited temperature range. The Society's Committee D-20 on Plastics has set up a tentative method for determining the permanent effect of heat on plastics. Further work along these lines is extremely important for the evaluation of plastic materials for structural applications.

TABLE I.—MOLDING CONDITIONS.

Material	Specimen	Mold Temperature, deg. Cent.	Pressure, psi.	Molding Time, min.
Wood-flour filled phenolic molding compound Specific gravity 1.35 to 1.37 (molded)	A.S.T.M. 5 by ½ by ½-in. specimen (D 256 − 38)[a]	170	4000	10
	5 by 7 by 3/16-in. slab for A.S.T.M. tension specimen	170	3000	10
Pure phenolic resin compound Specific gravity = 0.96 to 0.98 (molded)	A.S.T.M. 5 by ½ by ½-in. specimen	170	4000	10
	A.S.T.M. Type 1. Tension specimen 0.150 in. thick (D 638 − 41T)[b]	170	6200	5—Cure 5—Cool[c]
Asbestos filled phenolic molding compound Specific gravity = 1.94 to 1.96 (molded)	A.S.T.M. 5 by ½ by ½-in. specimen	170	4000	10
	5 by 7 by 3/16-in. slab for A.S.T.M. tension specimen	170	7000	10
Cord filled phenolic molding compound Specific gravity = 1.35 to 1.37 (molded)	A.S.T.M. 5 by ½ by ½-in. specimen	170	4000	10
	5 by 7 by 3/16-in. slab for A.S.T.M. tension specimen	170	7000	15
Fabric filled phenolic molding compound Specific gravity = 1.35 to 1.37 (molded)	A.S.T.M. 5 by ½ by ½-in. specimen	170	4000	10
	5 by 7 by 3/16-in. slab for A.S.T.M. tension specimen	170	7000	15

[a] Standard Methods of Test for Impact Resistance of Electrical Insulating Materials (D 256 − 38), 1939 Book of A.S.T.M. Standards, Part III, p. 252.

[b] Tentative Methods of Tension Testing of Plastics (D 638 − 41 T), 1941 Supplement to Book A.S.T.M. Standards, Part III, p. 457.

[c] Because of the difficulty in molding this type of material the mold was chilled before removing the specimen.

Because the physical properties of the molded material depend to a marked degree upon the technique used in the preparation of the materials and test specimens (15), a knowledge of this technique is essential if the results are to be properly evaluated.

The filled materials studied contained 50 per cent phenol-aldehyde resin and 50 per cent filler with the exception of the asbestos-filled material which contained 40 per cent resin and 60 per cent filler. This change was necessary to give a moldable composition.

The wood flour-filled and asbestos-filled molding compositions were prepared by rolling the resin and filler on a set of differential rolls in the usual manner for the preparation of general-purpose phenolic molding compounds. Three typical phenol-aldehyde resins were evaluated in connection with the wood-flour filler:

1. A typical one-stage, basic catalyzed resin.
2. A typical two-stage, acid catalyzed resin.
3. A semi-two-stage, dual catalyzed resin.

No appreciable difference in properties were exhibited over the temperature range, and so the two-stage resin was used in connection with the other fillers and for the preparation of the pure resin molding compound.

The high impact materials, macerated fabric, cord, and sisal fiber fillers were blended with the resin in a wet-mix process. Mixing was held to the minimum necessary to assure a properly blended material in order to prevent disintegration of the filler and assure the maximum strengths. The wet resin-filler mix was dried, and the resin polymerized further to give a moldable composition.

Table I give the molding conditions for the various materials. The variation in the pressures on the 5 by 7 by $\frac{3}{16}$-in. slabs was necessitated by the variations in the plasticity of the several molding compositions. All specimens were compression molded.

The tension specimens for all the filled materials were machined from 5 by 7 by $\frac{3}{16}$-in. slabs, following the requirements for type 1 test specimen in the A.S.T.M. Tentative Methods of Tension Testing of Plastics (D 638 – 41 T),[3] except for the 7-in. over-all length. The pure resin was molded in a compression mold of the type specified for producing type 1 samples according to Methods D 638. This was necessary since the 5 by 7 by $\frac{3}{16}$-in. slab could not be satisfactorily molded and machined of this material.

Impact and flexural test specimens, $\frac{1}{2}$ by $\frac{1}{2}$ by 5-in. bars, of the pure resin and asbestos and wood-flour filled material were molded in a 5-bar gang mold as specified in A.S.T.M. Tentative Specifications for Molds for Test Specimens of Molded Materials Used for Electrical Insulation (D 647 – 41 T).[4] The impact grade materials, cord, sisal fiber, and macerated fabric filled, were molded in a single bar mold specified in Specifications D 647 as is customary for this type of material. Dimensions were held to 0.512 ± 0.006 in. on the variable dimensions for all specimens.

All specimens were conditioned after molding, according to A.S.T.M. Tentative Methods of Preconditioning Plastics and Electrical Insulating Materials for Testing (D 618 – 41 T)[5] for 48 hr. at 50 ± 3 C. (122 ± 5.4 F.), and placed in a desiccator after removal from the oven. Prior to testing, the specimens were conditioned at the desired temperature for from 4 to 5 hr. Tests were run immediately upon removal of the test specimen from the conditioning chamber.

Impact data were obtained on a Tinius-Olsen impact machine with a capacity of 100 in-lb. ($8\frac{1}{3}$ ft-lb.), and on a Baldwin-Southwark impact tester (developed by the Bell Telephone Laboratories) having a capacity of 4.0 ft-lb. Tests were run according to A.S.T.M. Standard Methods of Test for Impact Resistance of Electrical Insulating Materials (D 256 – 38),[6] using the notched Izod method in which the stress was

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[3] 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 457.

[4] *Ibid.*, p. 317.

[5] *Ibid.*, p. 320.

[6] 1939 Book of A.S.T.M. Standards, Part III, p. 252.

TABLE II.—TENSILE STRENGTH (PSI.) *Versus* TEMPERATURE.[a]

Material	−80 C.	−25 C.	25 C.	80 C.	120 C.	150 C.	230 C.
Cord filled phenolic	6210 ± 610 (n = 5)	6340 ± 330 (n = 5)	6020 ± 400 (n = 5)	4830 ± 510 (n = 5)	4300 ± 500 (n = 5)	4220 ± 400 (n = 4)	3280 ± 300 (n = 5)
Fabric filled phenolic	6430 ± 340 (n = 8)	6370 ± 230 (n = 8)	6120 ± 260 (n = 7)	5080 ± 180 (n = 10)	4430 ± 220 (n = 9)	3950 ± 400 (n = 5)	....
Wood-flour filled phenolic	6280 ± 230 (n = 30)	6490 ± 300 (n = 28)	6100 ± 270 (n = 33)	4770 ± 180 (n = 34)	4430 ± 170 (n = 36)	3750 ± 270 (n = 38)	....
Unfilled phenolic resin compound	5150 ± 600 (n = 4)	5790 ± 1040 (n = 5)	6200 ± 400 (n = 4)	5200 ± — (n = 3)	....	....	....
Asbestos filled phenolic	6670 ± 430 (n = 4)	5210 ± — (n = 3)	5570 ± 730 (n = 4)	5500 ± — (n = 2)	....	5150 ± — (n = 1)	5170 ± — (n = 2)

[a] The number of specimens, n, broken at each temperature is given below each strength figure. The average for the stated number of specimens falls within their given limits 9 times in 10.

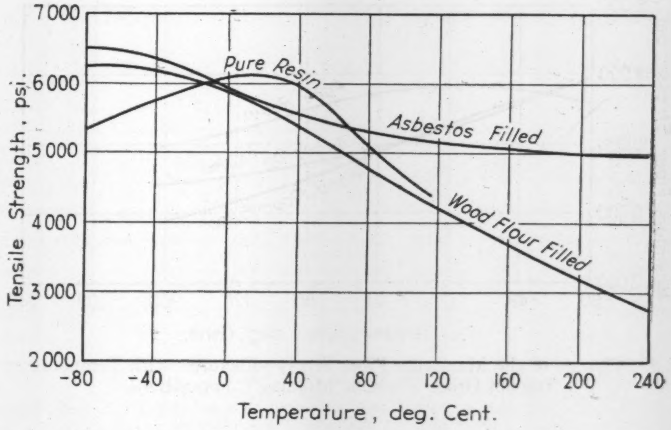

FIG. 1.—Change of the Ultimate Tensile Strength with Temperature for Low Impact Phenolic Molding Compositions.

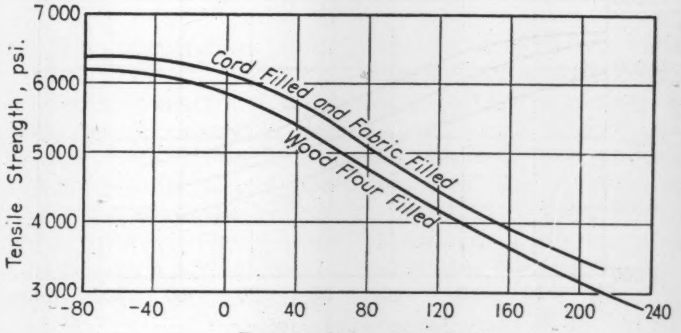

FIG. 2.—Change of the Ultimate Tensile Strength with Temperature for Impact Grade Phenolic Molding Compositions

TABLE III.—FLEXURAL STRENGTH (PSI.) *Versus* TEMPERATURE.[a]

Material	−80 C.	−25 C.	25 C.	80 C.	120 C.	150 C.	180 C.
Cord filled phenolic	15 700 ± 880 (n = 10)	15 900 ± 1800 (n = 7)	12 900 ± 460 (n = 10)	11 800 ± 670 (n = 8)	9 720 ± 930 (n = 8)	9 200 ± 700 (n = 7)	8560 ± 730 (n = 7)
Fabric filled phenolic	14 800 ± 730 (n = 10)	12 400 ± 540 (n = 8)	12 000 ± 570 (n = 10)	9 500 ± 450 (n = 10)	8 010 ± 400 (n = 10)	7 330 ± 700 (n = 10)	....
Wood-flour filled phenolic	13 100 ± 650 (n = 40)	11 700 ± 550 (n = 40)	9 770 ± 460 (n = 40)	7 880 ± 550 (n = 40)	6 200 ± 530 (n = 40)	5 380 ± 620 (n = 40)	....
Unfilled phenolic resin compound	12 300 ± 890 (n = 10)	12 400 ± 950 (n = 10)	13 300 ± 960 (n = 10)	12 300 ± 880 (n = 9)	10 500 ± 980 (n = 8)	10 000 ± 820 (n = 8)	....
Asbestos filled phenolic	13 700 ± 600 (n = 10)	11 900 ± 640 (n = 10)	10 200 ± 670 (n = 10)	9 320 ± 770 (n = 10)	8 580 ± 330 (n = 10)	7 260 ± 780 (n = 10)	....

[a] The number of specimens, n, broken at each temperature is given below each strength figure. The average for the stated number of specimens falls within their given limits 9 times in 10.

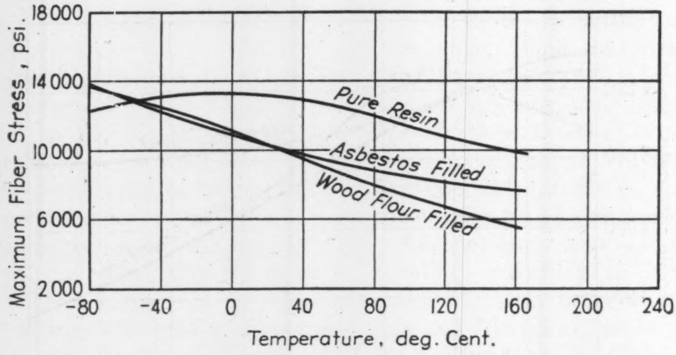

FIG. 3.—Change of the Maximum Fiber Stress—Flexure—with Temperature for Impact Grade Phenolic Molding Compositions.

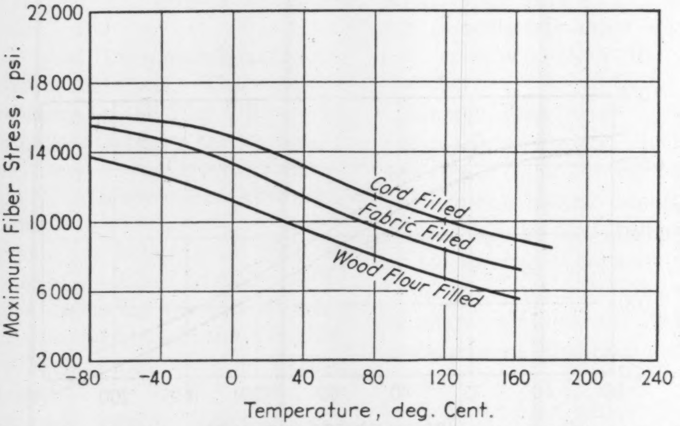

FIG. 4.—Change of the Maximum Fiber Stress—Flexure—with Temperature for Impact Grade Phenolic Molding Compositions.

applied perpendicular to the direction of the molding pressure.

Tensile and flexural data were obtained on a Tinius Olsen Universal Tester, a screw-type machine with constant rate of crosshead motion, and 10,000-lb. range. The machine was equipped with an insulated housing which enclosed the tension grips or flexural supports and the specimen. Temperatures from 60 C. to 225 C. (140 F. to 437 F.) were obtained by circulating dry air over heaters enclosed in the cabinet. For temperatures from 25 C. to −80 C. (77 F. to −112 F.) dry air, precooled by contact with solid carbon dioxide, was circulated in the cabinet. Tension tests were run according to A.S.T.M. Methods D 638 using the type 1 specimen with a thickness of 0.1875 ± 0.008 in. Flexure tests were run according to A.S.T.M. Standard Methods of Testing Molded Materials Used for Electrical Insulation (D 48 - 39).[7]

Tensile Properties

Data showing the change of tensile strength with temperature are given in Table II and are graphically presented in Figs. 1 and 2. The tensile strengths for the filled materials decrease with an increase in temperature over the entire temperature range. Within the limits of experimental error, the three materials containing organic fillers (wood flour, cord, and fabric) have identical strengths at the various temperatures. The asbestos-filled material stands up somewhat better at the higher temperatures and between 160 C. to 200 C. (320 F. to 390 F.) is roughly 25 per cent higher in tensile strength than the other compositions. The pure resin curve is unique in that it shows a maximum value at 25 C. (77 F.) which is about 15 per cent above the strength of the wood flour filled at the same temperature. Above 25 C. the strength of the pure resin falls off until at 80 C. (175 F.) it is the same as the organic filled compositions. At temperatures below 25 C. the strength drops and at −80 C. (−112

TABLE IV.—IMPACT STRENGTH (FT-LB. PER IN. OF NOTCH) *Versus* TEMPERATURE.[a]

Material	−80 C.	−25 C.	25 C.	80 C.	120 C.
Cord filled phenolic	4.85 ± 0.35 (*n* = 18)	5.50 ± 0.43 (*n* = 18)	7.21 ± 0.42 (*n* = 20)	8.57 ± 0.64 (*n* = 18)	10.05 ± 0.56 (*n* = 18)
Fabric filled phenolic	1.61 ± 0.12 (*n* = 10)	2.01 ± 0.22 (*n* = 10)	2.37 ± 0.28 (*n* = 10)	2.95 ± 0.23 (*n* = 10)	3.11 ± 0.25 (*n* = 10)
Wood-flour filled phenolic	0.29 ± 0.006 (*n* = 80)	0.32 ± 0.010 (*n* = 80)	0.31 ± 0.016 (*n* = 80)	0.31 ± 0.006 (*n* = 80)	0.32 ± 0.006 (*n* = 80)
Unfilled phenolic resin compound	0.269 ± 0.005 (*n* = 12)	0.260 ± 0.02 (*n* = 12)	0.248 ± 0.01 (*n* = 12)	0.233 ± 0.01 (*n* = 20)	0.224 ± 0.01 (*n* = 17)
Asbestos filled phenolic	0.300 ± 0.006 (*n* = 18)	0.278 ± 0.006 (*n* = 18)	0.284 ± 0.01 (*n* = 16)	0.283 ± 0.008 (*n* = 16)	0.282 ± 0.01 (*n* = 16)

Material	150 C.	170 C.	200 C.	220 C.	250 C.
Cord filled phenolic	11.1 ± 0.46 (*n* = 18)	9.94 ± 0.61 (*n* = 18)	10.6 ± 0.51 (*n* = 18)	2.47 ± 2.1 (*n* = 18)	.....
Fabric filled phenolic	3.08 ± 0.24 (*n* = 10)	2.68 ± 0.40 (*n* = 10)	0.99 ± 0.21 (*n* = 10)	.....	.....
Wood-flour filled phenolic	0.33 ± 0.006 (*n* = 80)	0.31 ± 0.02 (*n* = 80)	0.25 ± 0.02 (*n* = 80)	.....	.....
Unfilled phenolic resin compound	0.225 ± 0.01 (*n* = 19)	0.230 ± 0.01 (*n* = 18)	.....	.....	0.222 ± 0.01 (*n* = 18)
Asbestos filled phenolic	.....	0.293 ± 0.01 (*n* = 16)	0.288 ± 0.02 (*n* = 8)	.....	0.286 ± 0.01 (*n* = 16)

[a] The number of specimens, *n*, broken at each temperature is given below each strength figure. The average for the stated number of specimens falls within their given limits 9 times in 10.

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[7] 1939 Book of A.S.T.M. Standards, Part III, p. 244.

F.) is about 20 per cent of that of the other materials.

FLEXURAL PROPERTIES

Figures 3 and 4 and Table III present data on the change of flexural strength with temperature. The curves for wood-flour, fabric, and cord-filled molded compositions are parallel and show practically a linear decrease in flexural strength with increasing temperature. The asbestos filled also shows a decrease in strength with increasing temperature, but again withstands the higher temperatures better than the organic filled compositions. The data for flexural strength of the unfilled resin are surprising in two respects:

1. They are higher than expected, about 25 per cent above that of the wood flour filled.
2. They fall off slightly below 25 C. instead of continuing to increase as do the filled materials.

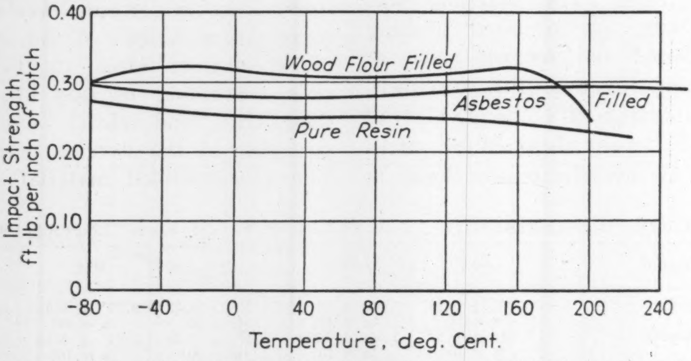

FIG. 5.—Change of the Notched Izod Impact Strength with Temperature for Low Impact Phenolic Molding Compositions.

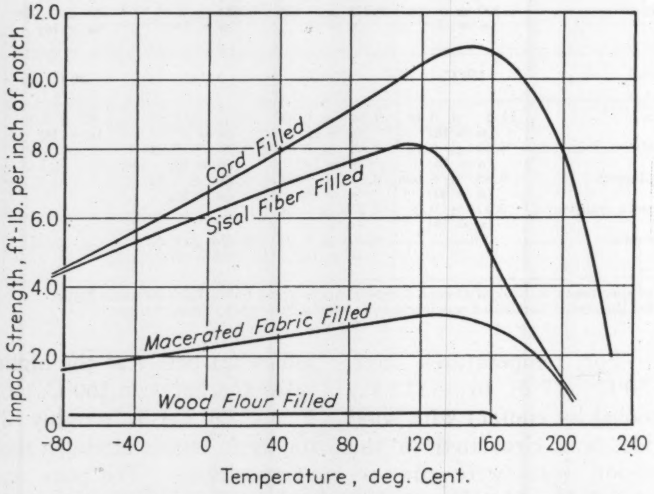

FIG. 6.—Change of the Notched Izod Impact Strength with Temperature for Impact Grade Phenolic Molding Compositions.

Impact Properties

The effect of temperature on the notched Izod impact strength is given in Table IV and Figs. 5 and 6. For the high impact materials, cord, sisal fiber and macerated fabric filled, the strength increases with increasing temperature to a critical point and then falls off sharply. The critical point occurs at from 160 to 170 C. (320 to 340 F.) and is accompanied by charring of the organic filler. The wood-flour filled material exhibits relatively little change of impact strength with temperature up to a critical point at about 160 C. (320 F.), after which the strength falls off sharply. The strength of the pure resin and asbestos filled compositions remains practically constant over the temperature range investigated.

Reliability of Data

The impact values recorded in the attached tables represent the average of from 10 to 20 individual observations. In the case of flexural and tensile strength values, from 5 to 11 observations were made for each value reported.

However, in running the tension tests on the asbestos filled and pure resin compositions, many of the specimens broke in the grips before the ultimate strength was reached. Consequently, only a few test results were available for many of the conditions reported. These data are, therefore, only preliminary, and are presented here to show the trend of results obtained to date.

The limits within which the observed value may be expected to lie 9 times in 10, limits of uncertainty, were calculated according to the A.S.T.M. Manual on Presentation of Data[8] for the data presented. These limits are included in Tables II, III, and IV and are approximately 6 per cent for flexure, 4 per cent for impact, and 5 per cent for tension tests. The percentage variation in strengths from batch to batch of the same material is of the same order of magnitude.

The data presented for the wood-flour filled phenolic is the average of results on four typical molding compositions, differing only in the basic resin formulation. Results on the individual materials exhibited no appreciable differences in properties over the range investigated.

It should be noted that the notched Izod impact test may be misleading in certain cases where the impact strength of the materials under test is low, and where the specific gravities of different molded materials are substantially different. In low impact materials the energy required to throw the broken end of the test specimen becomes a significant portion of the energy expended, and is dependent on the specific gravity of the molded specimen. For example, the asbestos-filled phenolic (specific gravity, 1.95) gives a standard impact value which compares favorably with wood-flour filled molded compositions (specific gravity, 1.36) and pure resin compositions (specific gravity, 0.96). However, the behavior of these materials in machining and general handling indicates that asbestos-filled materials have less shock resistance than either of the other two.

Practical Importance

Over the temperature range of normal use, −50 C. to 70 C. (−60 F. to 160 F.), the mechanical strengths of phenolic molding compositions deviate from the value at 25 C. (77 F.) by about ± 15 per cent. The tensile and flexural strengths show a decrease with increasing temperature. This is offset in the high impact materials by an increase in impact strength with increasing temperatures.

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[8] Issued as separate publication.

Tensile properties are practically independent of the filler used. The use of cord or macerated fabric, high impact fillers, improves the flexural strengths slightly over the temperature range. Impact strengths may be greatly increased over the entire temperature range by use of fibrous fillers such as macerated fabric, sisal fiber, cord, etc. (**16, 17**).

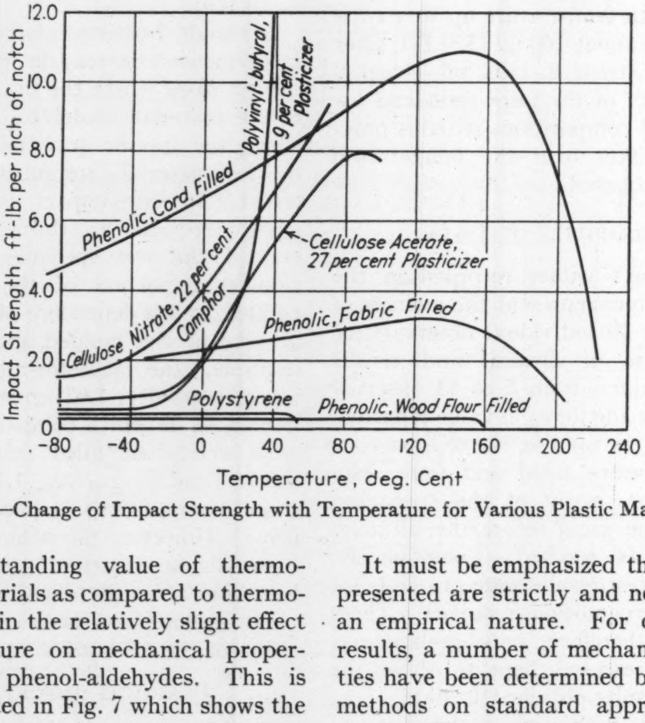

FIG. 7.—Change of Impact Strength with Temperature for Various Plastic Materials.

The outstanding value of thermosetting materials as compared to thermoplastics lies in the relatively slight effect of temperature on mechanical properties of the phenol-aldehydes. This is well illustrated in Fig. 7 which shows the change in impact strength with temperature for various plastic materials (**18, 19**). Comparing the wood-flour filled phenolic composition with injection-molded polystyrene it may be seen that the critical point is roughly 100 C. (212 F.) higher for the phenolic. The maximum temperature of serviceability for thermoplastics ranges from 40 C. (105 F.) for polyvinyl-butyral containing 9 per cent plasticizer to 90 C. (195 F.) for cellulose acetate containing 27 per cent plasticizer. The high impact phenolic materials are serviceable at temperatures as high as 160 to 170 C. (320 to 340 F.). At temperatures below 25 C. (77 F.) the impact strengths of cellulose acetate, cellulose nitrate, and polyvinyl-butyral fall off sharply, giving values less than 50 per cent that of the cord filled phenolic.

It must be emphasized that the data presented are strictly and necessarily of an empirical nature. For comparative results, a number of mechanical properties have been determined by A.S.T.M. methods on standard approved specimens. The results obtained give data for similar materials tested under identical conditions. It is often difficult to use the actual numerical values in evaluating a specific material for a particular application, but these values do serve as a basis for selecting the most suitable material. Since the form factor, mold design, molding conditions, treatment following molding, and numerous other factors affect the strength of the molded article, the final evaluation of the material should be based on actual service tests on the finished product.

Molded phenolic compositions, particularly those of the higher impact type, possess mechanical properties which make them practical as materials for use in structural applications.

REFERENCES

(1) T. S. Carswell, "Plastics in the Present Emergency," *Chemical Industries*, Vol. 48, February, 1941, p. 219.

(2) Duncan Campbell, "Resinoid Equipment in Wartime," *Chemical and Metallurgical Engineering*, Vol. 48, pp. 90–93 (1941).

(3) Jacobi and Thum, "Mechanische Festigkeit von Phenol-Formaldehyde, Kunststoffen," *Forschungshief*, Vereines Deutscher Ingenieure, May/June, 1939, p. 396.

(4) N. A. de Bruyne, "Plastic Materials for Aircraft Construction," *Journal*, Royal Aeronautical Soc., Vol. 41, pp. 523–590 (1939).

(5) Peter Genung, "Long-Time Creep of Plastics, Plastic Trends," Vol. 2, No. 3, February 1, 1942, p. 7, Plastics Institute.

(6) H. W. Mellville, "Plastics in Industrial Physics," *Reports in Progress on Physics*, Vol. V (1938).

(7) H. N. Haut, "Synthetic Resins in Construction," *Aviation*, Vol. 41, March, 1942, p. 84.

(8) H. Perkuhn, "The Creep of Laminated Synthetic Resin Plastics," Translated as *Technical Memo No. 995*, National Advisory Committee on Aeronautics (1941).

(9) W. Hofinghof, "Plastics in the German Railway System," *Kunststoffen*, Vol. 31, pp. 1–10 (1941).

(10) Herbert Chase, "Plastics or Metals," *British Plastics*, Vol. 13, pp. 68–71 (1941).

(11) J. Delmonte and W. Dewar, "Factors Influencing Creep and Cold Flow of Plastics," ASTM BULLETIN, No. 112, October, 1941, pp. 35–41.

(12) R. Nitsche and E. Salewski, "The Effect of Temperature on the Strength of Plastic Materials," *Kunststoffe*, Vol. 29, pp. 209–220 (1939).

(13) T. T. Oberg, R. T. Schwartz, and D. A. Shinn, "Mechanical Properties of Plastic Materials at Normal and Subnormal Temperatures," *Air Corps Technical Report No. 4648*, June 6, 1941.

(14) S. W. Place, "Effect of Heat on Phenolic Laminates," *Modern Plastics*, Vol. 18, pp. 59–62 (1941).

(15) F. Grush, "Impact Resistance as a Function of Cure," *Plastics Institute Research Project X-115, Plastic Trends*, Vol. 2, No. 5, March 1, 1942, p. 7.

(16) C. H. Whitlock and R. U. Haslanger, "High Impact Strength Molding Compositions," *Modern Plastics*, Vol. 19, June, 1942, pp. 70–71.

(17) V. Meharg, "Why Fillers in Thermosetting Molding Materials," *Modern Plastics*, Vol. 15, pp. 46–54 (1937).

(18) T. S. Carswell, R. F. Hayes, and H. K. Nason, "Physical Properties of Polystyrene as Influenced by Temperature," *Industrial and Engineering Chemistry*, Vol. 34, pp. 454–457 (1942).

(19) T. S. Lawton, Unpublished Data, Plastics Division, Monsanto Chemical Co.

(20) E. T. McBride, J. B. Lunsford and A. Philipson, "Some Views on Plastics in Wartime," *Modern Plastics*, Vol. 19, April, 1942, pp. 39–45.

(21) S. S. Kistler, "The Thermoplastic Behavior of Linear and Three-Dimensional Polymers," *Journal of Applied Physics*, Vol. 11, pp. 760–778 (1940).

(22) P. M. Kozlov, "Physical Effect of Temperature on Tensile Strength of Texolite," *Trudy Sessii Akad. Nauk Org. Khim.*, pp. 91–97 (1939).

DISCUSSION

Mr. W. N. Findley.[1]—The authors have done a thorough job of studying the effect of different temperatures on the results of certain mechanical tests of phenolic materials. There remains, however, a considerable amount of work to be done before complete knowledge of the effect of temperature on the mechanical properties of these materials is available. The effect of temperature on the fatigue strength of these materials is not as yet completely known; and nothing is known about the effect of temperature on creep characteristics.

Then there is another very important but largely unexplored field—the effect of changes in moisture content (as a result of humidity changes) on the results of all the mechanical tests. While the moisture absorption of phenolic materials is relatively small, it may be that even small amounts of moisture may produce a large change in creep characteristics or fatigue strength.

Mr. R. H. Spry.[2]—Will the author explain the mechanics of obtaining the low temperatures as well as the high temperatures for the various mechanical tests which were made? There has been an increased demand for wider temperature ranges in material testing and a specific description of the authors' procedure would be appreciated.

Mr. H. F. Moore.[3]—Were the impact specimens notched?

Mr. H. K. Nason.[4]—I should like to point out first that in presenting the paper I was doing so on behalf of the authors and all credit for the quality of it goes to them.

To answer Mr. Moore's question, the test specimens were all notched.

In reply to Mr. Spry's question, the samples were conditioned according to A.S.T.M. Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 – 41 T),[5] that is, 48 hr. at 50 C., followed by storage in a desiccator until used. These were then—in the case of the impact specimens—chilled at the low temperature desired or placed in an oven at the high temperature desired, removed therefrom and broken as fast as they could be clamped in the impact machine. This required about 4 or 5 sec. In the case of tension and flexural tests, a specially constructed housing was placed on the universal test machine completely surrounding the tension grips, or flexural support. This housing was maintained at the desired temperature by passing dry air either over heating coils or over carbon dioxide ice, temperature control being effected by suitable adjustment of the heating or cooling medium and the rate of air flow. The samples were preconditioned at the desired temperature for several hours before actually breaking.

To reply to Mr. Findley's question: the authors have pointed out that these samples were dried, and the air used, either hot or cold, was dry; hence the effect of moisture was minimized. It is realized that moisture is extremely important—perhaps not so much in the case of these phenolic materials as in the case of thermoplastics, such as cellulose acetate, and similar materials which are moisture susceptible. Additional work on the effect of moisture is in progress. This, of course, complicates work of this kind by multiplying the number of tests by the number of different moisture equilibrium states which must be tested.

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[1] Associate in Theoretical and Applied Mechanics, College of Engineering, University of Illinois, Urbana, Ill.

[2] Chief Engineer, Mica Insulator Co., Schenectady, N. Y.

[3] Professor of Engineering Materials, University of Illinois, Urbana, Ill.

[4] Assistant Director of Research, Plastics Division, Monsanto Chemical Co., Springfield, Mass.

[5] 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 320.

MESSRS. T. S. CARSWELL,[6] D. TELFAIR,[6] and R. U. HASLANGER[6] (*authors' closure, by letter*).—The authors wish to thank Mr. Nason for presenting the paper, and for contributing to the discussion.

We agree with Mr. Findley's statement to the effect that much work remains to be done in the field of temperature and humidity testing of plastics. The authors' investigation of the effect of prolonged heating on the strength of phenolic materials (results to be published soon) represents another contribution to our limited knowledge of the properties of phenolic materials. Also, work is now in progress on the problem of creep, and of its dependence on temperature and humidity. Such work is, of necessity, tedious and slow, and it is hoped that other laboratories will be able to supplement such data as we may obtain.

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[6] Research Director, Research Physicist and Research Chemist, respectively, Plastics Division, Monsanto Chemical Co., Springfield, Mass.

METHOD FOR INSPECTION AND CONTROL OF LIQUID STARCH-BASE ADHESIVES FOR BOTTLE LABELING AND CASE SEALING

BY A. HERMAN[1] AND F. M. KNOWLTON[1]

SYNOPSIS

A procedure has been developed by which the starch-base adhesives for bottle labeling and case sealing can be controlled. The viscosity of the sample to be tested was measured by a falling-ball type viscometer. Adhesive films were spread with a Bird applicator and examined for hygroscopicity, color, plasticity, and adhesive strength. Moisture, alkalinity or acidity, and ash were determined as chemical properties.

The film examination described was found to be helpful in selecting adhesives for various applications. The film characteristics along with the viscosity and chemical tests were used to examine new shipments for uniformity and to control their application.

Liquid starch adhesives have found widespread use in the packaging industry for bottle labeling and case sealing and in the fabrication of paper containers. In contrast to the wide acceptance of these products by industry, very little work has been done to develop methods to determine their appropriateness for certain applications and to control their uniformity. Many industries still must resort to the "trial-and-error" method of testing if the suitability of a new type or brand of adhesive is to be investigated. While this method is sufficient in testing the actual application performance, it is not satisfactory in that nothing can be predicted regarding actual adhesive power until several months or perhaps a year has elapsed. Also, quite often when the trial-and-error method is employed to carry out adhesive experiments, there is a tendency to place more emphasis upon application rather than upon ultimate adhesive power.

In order to minimize the risks taken in trying out adhesives of unknown quality and composition on high-speed production lines, the following plan of testing was adopted: The quality of the product was first established by thorough laboratory tests to make certain that there was little danger of adhesive failure after finished packages had been distributed to wholesale and retail dealers. If this requirement was met, extensive plant tests involving quantities not less than one standard 50 to 60-gal. drum were carried out to be sure that it was adapted to all various methods of application. At this point several finished packages were taken as samples and stored at room temperature for observation over a period of several months. Whenever possible, even more extensive plant tests covering several months were run in order to disclose any undesirable effects which might result if decomposition should occur during storage. No adhesive was ever approved for general use until at least a drum of it had been run and proven satisfactory by a plant test.

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[1] Assistant to Vice-President, and Chemical Engineer, respectively, Joseph E. Seagram and Sons, Inc., Louisville, Ky.

An adhesive which passes a laboratory analysis will not give the best performance if its handling is neglected. Accordingly, after observing existing plant conditions for a period of time, adhesive mixing and handling was placed on a scientific basis. The need for control of mixing was made apparent when the conditions existing at the bottling plants were explored. The consistency was only guessed by crude methods such as observing its flow off a wooden paddle, by the finger test, etc. Due to these crude methods of control, the viscosity of the adhesives used varied and the product did not always give the best performance. The development of new products probably was also impaired because there was no reliable basis for any comparison.

The method of laboratory examination adopted was developed primarily for checking liquid starch products used for labeling liquor bottles and for sealing the finished cases for shipment. Though these two starch products represent only a small part of the field of adhesives now being used, many of the tests employed for them can be easily extended to cover other types of products. (In fact the resin and casein-base adhesives used to affix the strip stamps on our bottles are being checked by the analytical procedure described here.)

PROCEDURE

Sampling:

A regular pieced tinware milk stirrer with its handle lengthened to 36 in. was used to sample the liquid products which were received in 50 to 60-gal. steel drums. Because vegetable adhesives are subject to decomposition it is essential that a sampler, such as a milk stirrer, be used since it can be easily cleaned and sterilized. The contents of the drum to be sampled were first stirred to uniform consistency by using the sampler as a stirring device. The sample was then taken at random over the surface of the liquid, and was poured directly from the sampler bowl into a 1-qt. glass container which was

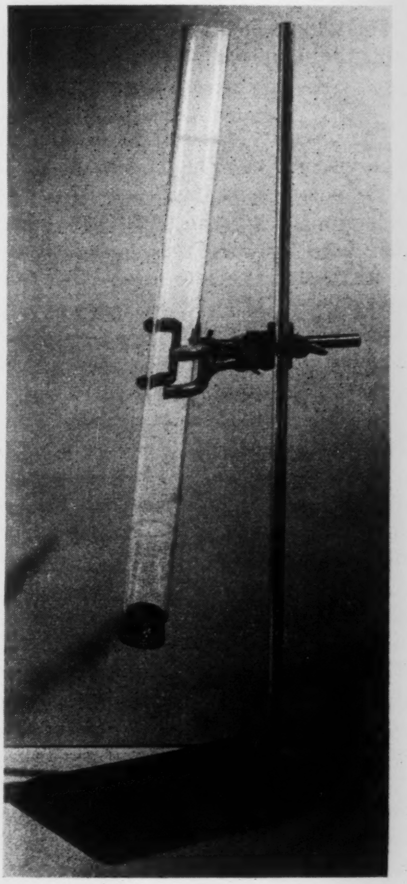

FIG. 1.—Viscometer.

closed immediately. When extremely viscous materials were sampled, only a small volume of the liquid along the vertical axis of the drum was agitated before the sample was taken.

Odor:

The odor was checked by observation while the sample was being taken.

Viscosity:

The viscosity was run on a falling ball type viscometer (Fig. 1), an adaptation of the commercial Hoeppler instrument, and similar to the A.S.T.M. instrument used for checking the consistency of nitrocellulose solutions. The tube was of Pyrex glass, 18 in. long, and 1.22 in. in inside diameter. Two rings were etched on the outside of it exactly 12 in. apart and equidistant from each end. The tube was supported at an angle of 10 deg. from vertical on a ring stand by a laboratory clamp. The clamp and holder were welded together to make sure that the inclination of the instrument was constant. The lower end of the tube was closed with a No. 7 rubber stopper. Glass spheres, 0.62 in. in diameter, weighing 5.39 ± 0.05 g., and standard $\frac{3}{4}$-in. (0.750 in. in diameter) steel ball bearings, chromium plated, weighing 28.175 ± 0.005 g. were used, respectively, for the light and heavy materials. A wire loop with a handle bent perpendicular to the plane of the loop was used to release the spheres in the viscometer tube.

Approximately 400 g. of sample were weighed into a tared 1-qt. glass vessel. In the case of very viscous products, some dilution was necessary. The dilution chosen for any particular product was the least dilution which would yield reproducible results on the instrument. In all cases sufficient diluent was added so that the viscosity was not more than 80 sec. The correct amount of water to add for any particular product was obtained by experimentation.

The required amount of water by weight for dilution was calculated, and a thorough mix was obtained by adding it very slowly while mixing with a stirring rod about $\frac{1}{4}$ in. in diameter. The sample preparation was brought to 25 C. by heating or cooling in a water bath with occasional mixing with a stirring rod to assure uniform temperature throughout the mixture. This preparation at 25 C. was poured slowly into the dry viscometer tube which had been immersed in a 25 C. water bath until ready for use. The tube was dried quickly by forcing paper towels through it and filled with liquid to about 1 in. from the top. A sphere, also cooled in the 25 C. water bath and dried, was introduced and gently lowered beneath the liquid surface with the wire loop. Care was taken to wet the sphere thoroughly so that no air bubbles would cling to it during its fall, otherwise inaccurate values will result. The sphere was then brought against the lower side of the tube, above the first mark, and released. The interval required for the center of the sphere to pass from the upper to the lower mark on the tube was timed by an accurate stop watch and was taken as a measure of the viscosity. In all cases an average value for the viscosity was obtained by dropping at least three spheres through the liquid being tested.

Mixing Properties:

About 200 g. of sample were placed in a 400-ml. beaker and mixed with a 600-rpm. model "V" Lightnin mixer equipped with a single three-blade propeller. The tendency for the adhesive to pull into the propeller—a measure of cohesiveness—was noted. Foaming tendencies were also observed at this point. A small amount of water was added with continued mixing and the ease of incorporation of diluent and tendency to form lumps were noted.

Film Examination:

Adhesive films were spread on plate glass for examination with a Bird applicator machined to a depth of 0.003 in. In cases where it was desirable to spread films on paper stock because the adhesive in question would not adhere to plate glass, 0.003-in. brass stencils of various shapes and sizes cut out of 0.003-in. brass shim stock were used (Fig. 2). The desired size stencil was placed over the paper stock to be coated, a drop of adhesive was applied at one end of the cutaway section and spread with the side of a straight rimless test tube or a stirring rod of large diameter. When either method was employed to lay films, care was taken to perform all spreading operations with a reasonably constant speed (usually 1 to 2 in. per second (**3**)[2]) to obtain films of reasonably uniform thickness.

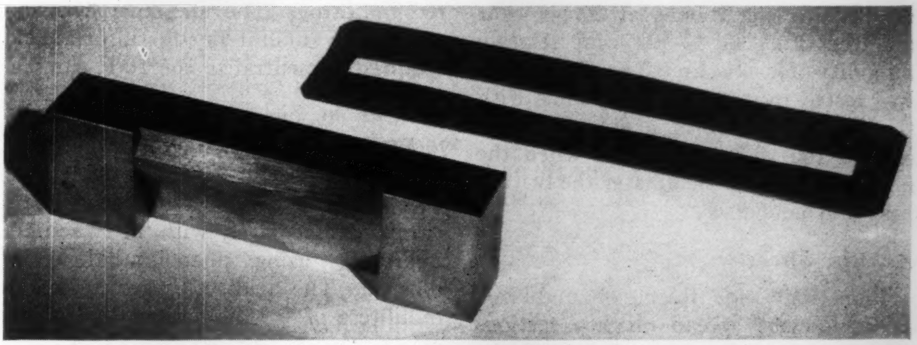
FIG. 2.—Bird Applicator and Brass Stencil.

The humidity chambers used for conditioning films for inspection as described below were simply medium and large size desiccators containing saturated salt solutions (**4**) to maintain the desired humidities. Both saturated ammonium chloride and saturated ammonium sulfate solutions were used to obtain a relative humidity set arbitrarily at approximately 80 per cent by our laboratory. Sulfuric acid solutions were used to maintain a 20 per cent relative humidity condition. The concentration of the sulfuric acid depended on the temperature at which the test was carried out.

Hygroscopicity:

Films of the undiluted sample were spread into 0.003-in. films on plate glass, dried for 24 hr. at room temperature, and then conditioned at 80 per cent relative humidity for 24 hr. After this treatment they were tested with the fingertip for excessive softening which indicates hygroscopicity.

In case this test showed an adhesive to be moderately hygroscopic and of doubtful quality an additional "performance test" was run. For instance, if the adhesive in question was a label gum, a 0.003-in. film of the gum was laid on a section of label which was then affixed to plate glass. If the section of label could not be removed without tearing after drying at room temperature for 24 hr. and being conditioned at 80 per cent relative humidity for 24 hr., its quality was considered satisfactory.

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[2] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 961.

Color:

Films of the undiluted adhesive were spread on plate glass, both dried at room temperature and conditioned at 20 and 80 per cent relative humidities for 24-hr. periods. The color, translucency or opacity were observed at these humidity extremes.

Brittleness:

A small well-mixed amount of the original sample was spread into 0.003-in. films on plate glass and dried at room temperature for 24 hr. One air-dried sample was conditioned at 20 per cent and the other at 80 per cent relative humidity for 24 hr. The brittleness was tested by scraping these films with the rounded point of a spatula. The shorter the ribbon produced when the film was scraped, the greater the brittleness and conversely.

Adhesive Strength:

Films 0.003-in. thick, of undiluted adhesive were spread on two sections of plate glass, dried at room temperature for 24 hr., and then one preparation conditioned at 20 per cent and the other at 80 per cent relative humidity for 24 hr. The relative resistance of the conditioned films to removal from the glass was taken as an indication of their final adhesive strength.

In the case of carton sealing adhesive which would not adhere to glass, similarly shaped sections of standard cartons were sealed together with 0.003-in. films of the undiluted adhesive laid with a stencil and allowed to dry at room temperature for 24 hr. Part of these preparations were conditioned at 20 per cent and the remainder at 80 per cent relative humidity for 24 hr. and were then torn apart to determine the adhesive strength. The quality of the adhesive was indicated by the percentage of the total film area still remaining intact after rupture of the joint.

Moisture:

The moisture content was determined by distillation with perchlorethylene using the distilling trap described by Langeland and Pratt (1).

Alkalinity:

A 10-g. sample of undiluted adhesive was mixed thoroughly with 100 ml. of warm distilled water (about 50 C.) in a beaker and titrated immediately with 0.2 *N* sulfuric acid using methyl red indicator. The alkalinity was expressed in milliliters of sulfuric acid required to neutralize the 10-g. sample.

Acidity:

A 10-g. sample of undiluted adhesive was mixed thoroughly in a beaker with 100 ml. of water at about 50 C. and titrated immediately with 0.2 *N* sodium hydroxide using phenolphthalein indicator. The acidity was expressed in milliliters of 0.2 *N* sodium hydroxide.

Ash:

Approximately 1 g. of sample was weighed into a tared platinum crucible which was heated gently until all volatile material was driven off. The remaining nonvolatile material was ignited to constant weight over a Meker burner.

DISCUSSION

Reproducible values for viscosity of starch products can be obtained only by following identical procedures of diluting and mixing for each successive determination. Erratic viscosity values, also discussed elsewhere (2, 5), are the result of varying degrees of thixotropic breakdown of the starchy materials.

The humidity tests were designed

primarily as a safeguard against crystallizing or hygroscopic materials. The humidities employed were intended to correspond to the extreme humidity conditions to which the adhesive was subjected in actual use.

"Tack" is defined as the pull resistance to splitting of a film of definite area and thickness. Preliminary tests indicate that initial tack is proportional to the viscosity regardless of the composition of the material. If it is verified that tack is proportional to viscosity, no tack test will be needed.

No method was developed for determining the drying time. Any procedure for this test must provide for constant temperature and humidity conditions, and a controlled rate of air flow over the films being tested. A method for spreading films of reproducible thickness must be employed, and if the films are to be laid on materials such as paper, the paper too must be standardized and humidified. However, predictions regarding the comparative drying time of various samples were made by considering the relative moisture content, viscosity, and deviation from chemical neutrality, particularly alkalinity.

Satisfactory specifications have been written for label and case sealing adhesives by adopting the above tests. As soon as any product has been proved to be satisfactory in regard to adhesive power by a laboratory test, and satisfactory in regard to application by a plant test, a mutual agreement was made with the supplier regarding the permissible variations of the various items such as viscosity, solids, alkalinity, etc., so that uniformity could be assured.

Acknowledgment:

We wish to thank the Arabol Manufacturing Co., National Adhesives, and the Paisley Products Co. for their cooperation and suggestions.

REFERENCES

(1) E. E. Langeland and R. W. Pratt, "Improved Trap for Moisture Determination by Distillation," *Industrial and Engineering Chemistry,* Vol. 10, No. 7, p. 401 (1938).

(2) R. Dulac and J. L. Rosenbaum, "Industrial Cold Adhesives," p. 16, J. B. Lippincott Co., New York, N. Y. (1937).

(3) H. A. Gardner, "Physical and Chemical Examination of Paints, Varnishes, Lacquers, and Colors," Ninth Edition, p. 100, Institute of Paint and Varnish Research, Washington, D. C. (1939).

(4) N. A. Lange, "Handbook of Chemistry," Fourth Edition, p. 1234, Handbook Publishers, Inc., Sandusky, Ohio.

(5) R. P. Walton, "A Comprehensive Survey of Starch Chemistry," Vol. 1, p. 209, The Chemical Catalog Co., New York, N. Y. (1928).

DISCUSSION

MR. M. LADEN[1] (*presented in written form*).—This presents a good opportunity to elaborate on the problems with which adhesive manufacturers are confronted and to outline the difficulties which have to be overcome to fill the various requirements of their customers.

In the distilling industry, two types of adhesives are generally used to adhere labels to bottles:

(*a*) Liquid dextrin-base adhesives
(*b*) Liquid starch-base adhesives

Both types have their definite place in labeling operations due to their different physical and chemical characteristics. Both have merits, but also some undesirable properties. Until there will be a way found to make label glues having only the good features of both types, users will have to compromise.

The ideal bottle-labeling glue should have the following characteristics:

1. Light color
2. High solid content
3. Strong initial tack
4. Good machining properties: free flowing, good spreading, nonfoaming, nonheavying up in the glue pot, noncottoning.
5. Fast setting
6. Good coverage

The dry glue film should be:

7. Transparent
8. Nonhygroscopic
9. Continuous
10. Of high tensile strength
11. Permanently flexible
12. Strongly adhering to glass and all kinds of label stock.

With respect to items 2 and 5, the higher the solid content of the glue, the less water has to evaporate to form the final film, and the faster it sets. As the water penetrates into the fibers on the glue side of the label, it causes them to swell and thus the label to curl. It is also evident that a glue having a higher solid content will shrink less by losing moisture than one with a lower solid content. The less a glue film shrinks when it dries out, the less it will crack, or, in case of a continuous film, the less it will cause permanent warping of the label to which it had been applied.

Tackiness.—As the initial tack is mainly dependent upon the viscosity of the glue and the viscosity again must be adjusted to the type of gluing machine and speed of operation, there are no other possibilities for the glue manufacturer than to offer a glue which has the highest viscosity still usable in a certain machine operation.

Working Qualities.—To apply the glue to one of the two surfaces to be glued together, machines are used where a horizontal metal roller rotates half submerged in the glue. The thickness of the glue film is regulated by a ductor blade. The glue must be fluid enough to flow freely to the roller and cover it evenly. There should not be foam formation which would cause overflowing of the glue pot or formation of pinholes in the glue film. When the transfer roller passes over the glue roller, the glue film tears and pulls some glue behind it. The cohesiveness of the glue should not be so great as to form fine hairlike threads or strings.

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[1] Chemical Engineer, The Arabol Manufacturing Co., Cicero, Ill.

The characteristics of the glue should be such that it is possible to spread the glue in an even film as thin as possible to give the best coverage. Besides it is an established fact that the strongest bond between two surfaces is obtained when the glue film is as thin as possible. A thick glue film acts as a filler between the two surfaces rather than as a binder.

Liquor bottles are distributed all over the country the year round and are subjected, therefore, to most adverse climatic conditions. In winter low temperatures will reduce the flexibility of the glue film and cause it to get more brittle and crack. In summer high humidity will cause the glue film to absorb moisture. The dried glue film should therefore possess enough flexibility not to become brittle in winter or at low humidities and it should not be hygroscopic to the extent that it would remoisten at high humidities. The tensile strength of the glue film should be greater under all climatic conditions than the strength of the paper fibers. When pulling the label from the bottle, the paper should tear, but the glue film should not come loose either from the glass or the label nor should the glue film itself be split.

Liquid Dextrin-Base Adhesives.—As explained in items 2 and 5, a high solid content is desirable. The average solid content of liquid dextrin-base bottle labeling glues is about 65 per cent. This is about the maximum concentration at which a highly soluble dextrin will give a solution of a viscosity usable in gluing machines.

The viscosity of straight dextrin solutions depends upon the solubility, the concentration, the temperature, and the type of dextrin used. Highly converted dextrines in which most of the starch granules are dextrinized are highly soluble and *vice versa*. By present methods it is difficult to convert all starch granules uniformly to dextrines without also forming dextrose. Dextrose has not only less adhesive power than dextrin, but increases also the hygroscopicity of the adhesive. By keeping the dextrose content of the dextrin as low as possible, the hygroscopicity of the dextrin adhesive can be kept within reasonable limits. A pure dextrin film will dry out to such an extent that it becomes brittle and chips off the glass at low humidities. Therefore a compromise is unavoidable and a certain amount of hygroscopicity has to be permitted to prevent crystallization of the dextrin film. The higher the solubility of a dextrin, the darker is its color generally. Here too we have to compromise.

Pure dextrin films are less flexible and less strong than gelatinized starch films. To improve the flexibility and film strength, it is therefore necessary to use dextrines which contain a certain amount of unconverted starch. This in turn causes a rise in viscosity. Therefore it is our endeavor to strike the happy medium where the amount of unconverted starch in the dextrin is just high enough to add some flexibility and toughness to the glue film without increasing the viscosity too much. This type of liquid dextrin base adhesive dries comparatively fast because of the relatively low amount of water present. It has therefore its place where springy labels should be adhered to round or oval shaped bottles where high initial tack and a minimum of warpage or blistering of the labels is of paramount importance.

Liquid Starch-Base Adhesives.—To maintain the inherent properties of gelatinized starch films in liquid starch-base adhesives as much as possible, the starch is not dextrinized but is modified so as to preserve many of the original starch properties. Such adhesives are characterized by their high viscosity, due to their thixotropic nature, and by the flexibility and toughness of the dry film. Because of the high viscosity of this type of glues, the solid content is limited to an average of 40 per cent. At this concentration the viscosity is still too high for proper machine operation and the original glue has to be diluted with water to a solid content of about 33 per cent. At this dilution the glue is not tacky enough to pull springy labels out of the magazine and can be used only on light or medium-weight labels. Due to the high percentage of water present in these glues at the working consistency, the glue film will dry much slower than one of a dextrin glue of the same viscosity. There will also be more warping of the labels. The dried glue film however is much superior in transparency, final adhesion, flexibility, and strength, to straight dextrin glues and the use of starch-base glues is well justified on lighter-weight labels.

Keeping in mind what has been said about the characteristics of the two types of label glues and their desirable and undesirable properties, it can readily be seen which tests are necessary to control these characteristics.

Tests for Dextrin-Base Label Glues.—The following tests are applied to dextrin-base glues:

(*a*) Viscosity
(*b*) Solids
(*c*) Dextrin and dextrose
(*d*) Starch
(*e*) Acidity
(*f*) Hygroscopicity
(*g*) Film color
(*h*) Film transparency
(*i*) Film hardness and elasticity
(*j*) Film strength (relative)
(*k*) Mixing and diluting properties
(*l*) Machining properties.

It is suggested to fill the dry viscosimeter tube with the glue, to close the upper opening with a rubber stopper and to submerge the tube in the water bath for as long as might be necessary to bring the glue inside the tube to a uniform temperature of 25 C., instead of bringing the tube and the glue separately to this temperature.

In testing for dextrin or starch (*c* and *d* above), a weighed portion of the glue sample, approximately 2 g., is transferred to a 400-ml. beaker, 200 ml. of distilled water are added and the glue is dispersed by stirring. A close-texture filter paper, such as Whatman No. 42, is dried at 105 C. to constant weight. The glue dispersion is then filtered through the weighed filter, placed in a Buechner funnel. The residue is washed thoroughly with distilled water at room temperature. Then the filter with the residue is transferred to a tared weighing bottle and dried for 3 hr. at 105 C. to constant weight. The weight of the residue is calculated to percentage of starch in the glue.

The percentage of dextrin and dextrose is calculated by subtracting the sum of the percentages of water and starch in the glue from 100.

Instead of using the fingertip to determine whether the film became tacky by absorbing moisture (*f* above), a piece of surgical cotton could be contacted with the glue film and pulled off again. The amount of fibers adhering to the film would indicate the degree of tackiness up to a point where an excessively hygroscopic film would become too soft to pull fibers.

The hardness (item *i*) could be tested by drawing lines on the conditioned film with Dixon's Eldorado pencils (17 degrees from 6B to 9H) starting with 6B. The hardness of the conditioned film could be expressed by the number of the pencil, which as the first in the row, does not leave a graphite mark on the film.

The degree of elasticity could be judged by the continuity of a film on glass, conditioned at 20 per cent relative humidity. Films of insufficient elasticity would show cracks or would chip off in flakes.

It is preferable to have the mixing and machining properties (items *k* and *l*) checked at the actual machining consistency than at another dilution ratio.

With all other testing methods for dextrin glues described in the paper, I am in full accord and have no comments.

Tests for Starch-Base Label Glues.—The following tests are applied to starch-base glues:

(*a*) Viscosity
(*b*) Solids
(*c*) Alkalinity
(*d*) Ash
(*e*) Hygroscopicity
(*f*) Film color
(*g*) Film transparency
(*h*) Film hardness and elasticity
(*i*) Film strength (relative)
(*j*) Mixing and diluting properties
(*k*) Machining properties.

Viscosity determinations of this type of adhesives are very unreliable because of their thixotropic nature. It is suggested therefore to leave out the viscosity test and check the uniformity of successive glue shipments by performing the other tests only.

The same suggestions are made as for items *f*, *i*, *k*, and *l* of Dextrin-Base Label Glues.

Case-SealingGlues.—Case-sealing glues are highly alkaline, liquid dextrin-starch-base adhesives.

The requirements for a perfect glue of this type are:

1. Good transfer of the glue from one flap to the other
2. Rapid setting
3. Quick drying
4. Good machining qualities (low viscosity of the diluted glue, not heavying up in the glue pot, not building up on the glue roller, nonfoaming, not skinning)
5. Good coverage

The dry film should be:

6. Of high strength
7. Not hygroscopic
8. Not crystallizing

After a glue film has been applied to the underside of the outer flaps, they are closed and pressed against the inner flaps by subjecting the case to pressure on a so-called compression unit. By contacting the two surfaces, enough glue should be transferred from the glued to the unglued surface to form an uninterrupted bond between them.

As the cases leave the compression unit, sometimes in as short a time as twenty seconds, rapid setting of the glue is imperative to prevent the flaps from popping open after the pressure is released. Part of the moisture and part of the highly soluble dextrin penetrate into the fibers of the board and a concentrated glue film remains on the surface. This film consists of less converted dextrin and starch particles which do not penetrate into the board because of their greater molecular size and consequent higher viscosity.

The soluble dextrin, anchored in the fibers by penetrating them, forms a strong bond with the film remaining on the surface. When this (already concentrated) film on the outer flaps is brought into contact with the inner flaps, another part of the moisture and of the soluble dextrin penetrates into the board of the inner flaps. Thus the glue film between the two surfaces should reach such a high concentration as to form an immediate bond and to dry quickly.

By varying the proportions of the more and the less soluble components of the glue, it is possible to control the rate of penetration and setting time within certain limits.

By diluting the glue with as little as about 10 per cent water, the originally rather viscous glue should become very fluid and machine perfectly without heavying up in the glue pot to give good coverage. If the viscosity of the glue would increase appreciably in the glue pot, either by excessive loss of moisture or by the mechanical agitation, the glue would build up on the glue roller. This would have the ill effect of poor glue transfer to the board. If the evaporation of water from the glue is too rapid, a skin will form on the top of the glue. By refilling the glue pot with fresh glue, this skin would break and form lumps. If these lumps should catch between the glue roller and the glue pot they would prevent an even film formation on the glue roller, and of course an interrupted film on the board. Excessive foaming would cause the glue pot to overflow.

The dried glue film should be strong enough to tear the fibers of the board stock when the flaps are pulled apart. It should not be hygroscopic so as to withstand high humidities without losing too much of its dry strength. It should also not become brittle to the extent to cause crystallization.

Control tests on case-sealing glues should comprise:

(*a*) Viscosity of the original glue
(*b*) Minimum amount of water necessary to give proper working consistency
(*c*) Solids
(*d*) Dextrin and dextrose
(*e*) Starch
(*f*) Alkalinity
(*g*) Ash
(*h*) Film hygroscopicity
(*i*) Film hardness and elasticity
(*j*) Film strength (relative)
(*k*) Mixing and diluting properties
(*l*) Machining properties

The same test methods could be applied as for the liquid dextrin-base label glues, with the exception that for test *h*, *i*, and *j*, films should be formed on board instead of on glass.

MR. B. E. KESTER[2].—There is one consideration I should like to present to the authors of this paper, and that is the problem of the permeability of a starch adhesive. On bottle labels with the adhesion of paper to glass, the permeation into the paper and not into the glass presents one set of conditions; in sealing compounds with adhesion of paper to paper, the permeation of the adhesive presents a second set of conditions.

In our work on starch adhesives we have found indications that permeability is a large factor in the adhesion of papers. How to measure the permeation and its effect by a quick control test would be desirable knowledge. In studies we have made on permeability, there is definite indication that bond and permeability are related. However, how to evaluate and control this factor we do not know.

MESSRS. A. HERMAN[3] and F. M. KNOWLTON[3] (*authors' closure*).—The discussion by Mr. Laden is very instructive in that it deals fundamentally in differentiating the various types and characteristic tests of adhesives included in the scope of the aforementioned subject. His suggested changes in testing procedures, such as, hygroscopicity by use of surgical cotton, film hardness by Dixon's Eldorado pencils, and determination of dextrin and dextrose, will be investigated.

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[2] U. S. Gypsum Co., Chicago, Ill.

[3] Assistant to Vice-President, and Chemical Engineer, respectively, Joseph E. Seagram and Sons, Inc., Louisville, Ky.

It is gratifying to see that Mr. Laden, representing the manufacturers of adhesives, in general agrees with the importance of testing the product with some accepted method. The reason for bringing the investigation before the A.S.T.M. was to stimulate further investigations among manufacturers and users.

I hope the A.S.T.M. finds the subject important enough to establish a committee dealing with this type of adhesive.

In reply to Mr. Kester, I may say that we have done no work on that point.

HYDRAULICALLY SUPPORTED SPHERICALLY SEATED COMPRESSION TESTING MACHINE PLATENS

By R. L. Templin[1]

Synopsis

Following a brief discussion of the problems involved in obtaining satisfactory round-end conditions in tests of columns, a new design of spherically-seated fixtures is described in some detail. Some results obtained in checking the efficacy of a particular set of such fixtures of 150 tons capacity are given, and certain features concerning the use of the apparatus are pointed out. From the evidence presented it is concluded that the new design offers definite advantages over previously available testing apparatus intended for similar uses.

In tests of round-end columns one of the most difficult conditions to attain is that wherein the ends of the specimen have no restraint in so far as rotation is concerned and no eccentricity of loading. Since in such tests it is necessary to transmit the load on a column through a mechanical joint of some sort, some frictional resistance to the tipping of the ends of the column may be expected. The problem presented is to keep this frictional resistance as low as possible, and at the same time provide the requisite degrees of freedom of motion.

Early attempts to obtain round-end conditions for columns of large size were made using plain spherical seats or pins. Test results reviewed by the Special Committee on Steel Column Research of the American Society of Civil Engineers indicated, in the important studies of column strength made between about 1880 and 1925, that the spherical seats and pins used gave a condition quite close to fixed ends.[2] In other words, the amount of friction was almost sufficient to prevent any rotation of the ends of the specimens. An improvement in the spherical seat type of fixture was made at the National Bureau of Standards[3] in 1924 by lining the spherical seat with small hardened steel balls. A set of similar testing machine fixtures was made at the Aluminum Research Laboratories in 1933 and tests of the apparatus indicated that the resistance to tipping under any load throughout its capacity range was indeed very small. The maximum capacity of the apparatus, however, was 25,000 lb., which permitted the testing of only small structural columns.

Other testing devices for obtaining the round-end condition include knife-edge fixtures and platens supported on ball or roller bearing trunions.[4] Usually these devices allow tipping about only one axis, although some that permit tipping in any direction have been built using intersecting knife-edges or intersecting trunions.[5] Examples of most of these various types of devices have been built and tried in the Aluminum Research Laboratories with varying degrees of success. Unfortunately the load capacities of such devices are usually small and the apparatus is subject to considerable elastic distortion during use. In addition, these particular devices do not permit rotation in a horizontal plane.

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[1] Chief Engineer of Tests, Aluminum Company of America, New Kensington, Pa.

[2] Progress Report of the Special Committee on Steel Column Research, *Transactions*, Am. Soc. Civil Engrs., Vol. 89, pp. 1526–1536 (1926).

[3] Tom W. Greene, "Strength of Steel Tubing Under Combined Column and Transverse Loading, Including Tests of Columns and Beams," *Technologic Paper No. 258*, Nat. Bureau Standards, May 23, 1924.

In general, compression tests of materials require greater total loads than tension tests of the same materials. Some of the reasons for this difference may be found in the types of specimens used and in the behavior of the various materials under the two kinds of loading. When testing compression specimens it may be desirable to load them as uniformly as possible throughout their cross-sectional area or to apply a load with a definite eccentricity. With the available testing machines and the dimensional variations involved in preparing the test specimens, uniform application of compression loads is very difficult without the use of a suitable load equalizing device. On the other hand, when it is desired to apply loads with a given amount of eccentricity, the usual types of fixtures are generally unsatisfactory.

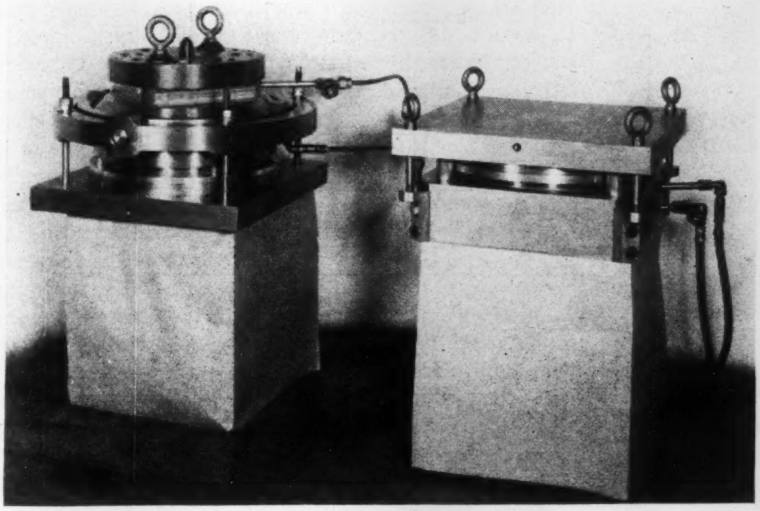
FIG. 1.—General View of the Compression Testing Apparatus.

Suitable fixtures for making round-end tests of column specimens, as well as for use in many other types of compression tests, therefore, (1) should have three degrees of freedom, that is, be able to rotate in any direction; (2) should possess as low resistance to rotation as possible; (3) should have relatively large total load capacity; (4) should distort a minimum amount during use; and (5) should permit the use of

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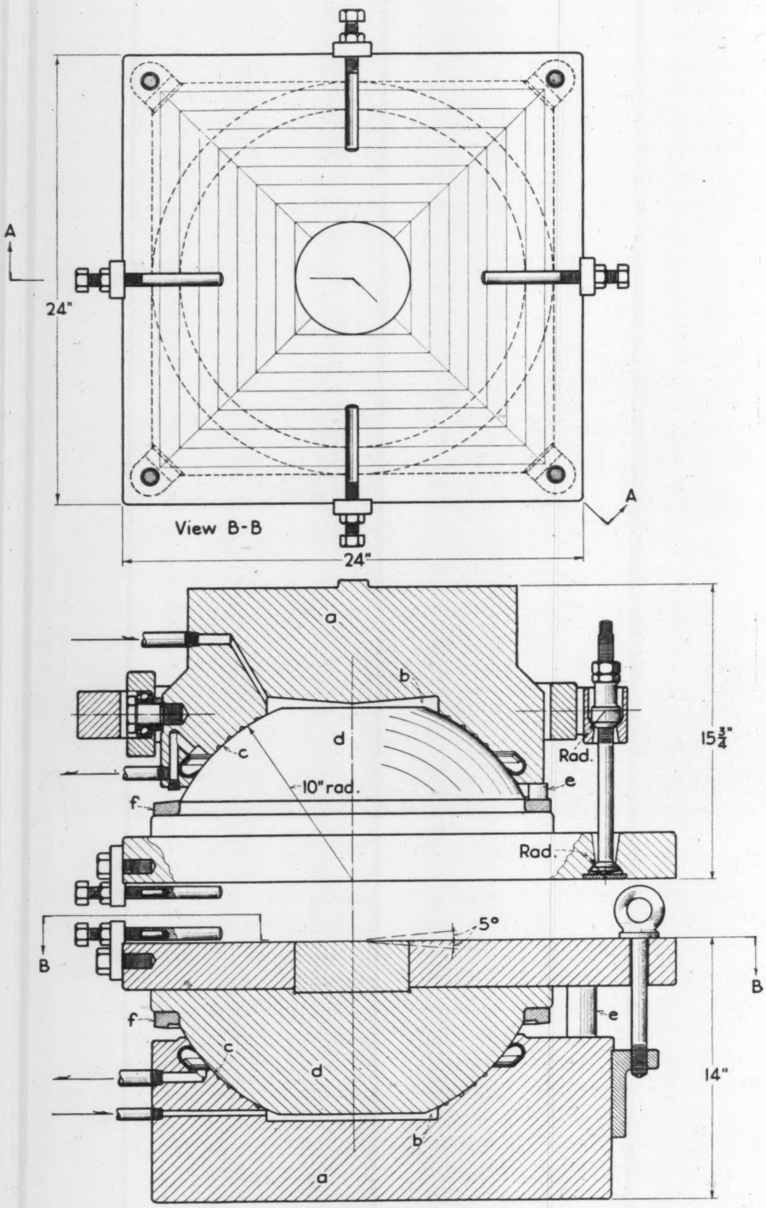
[4] H. W. Barlow, "A Fixture for Obtaining Pin-End Conditions in Column Testing," *Journal of Aeronautical Sciences*, Vol. 7, No. 2, December, 1939, pp. 72–74.

[5] H. W. Barlow, "The Column Strength of Closed, Thin-Walled Sections of 18–8 Stainless Steel," *Journal of Aeronautical Sciences*, Vol. 8, No. 4, February, 1941, pp. 151–161.


A
24"
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A
a
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10" rad.
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Rad.
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f
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14"
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a
Section A-A

FIG. 2.—Assembly Drawing of the Apparatus.

both uniform axial loads or loads with definite eccentricity. In addition to these primary requirements, such apparatus also should be readily adaptable to existing commercial testing machines, be readily handled, and reasonable in its cost.

Recently another attempt has been made to provide compression testing apparatus complying with the requirements just indicated. The resultant apparatus is shown in Figs. 1 and 2. Each fixture consists essentially of a supporting block *a*, in which a recess *b* has been provided for the supplying of oil pressure to the spherical bearing surface *c*, between the support *a* and the spherical part of the platen *d*. Oil is supplied at sufficient pressure to cause a continuous flow of a thin film between the spherical surfaces throughout the testing of a specimen. Provision is made for collecting the oil after its passage between the spherical surfaces, and returning it to the suction tank of the pump. The pump used with these fixtures is capable of supplying oil at varying pressures up to a maximum of about 5000 psi., depending upon the applied load. The total capacity of the fixture depends upon the diameter of the recess *b*, together with the maximum pressure exerted by the oil pump. In operation the oil pressure drops from the maximum value in the recess *b* to practically zero at the outer edge of the spherical contact area. An individual needle valve of suitable type is provided for metering the flow of oil to each support. These valves also serve to minimize, if not eliminate, oscillations of the platen arising from pump pulsations. Another valve is normally provided for controlling the oil supply of both platens simultaneously.

The annular grooves in the spherical seats of the supports *a*, constitute labyrinth packings and serve to insure a uniform thickness of the supporting oil film throughout the spherical areas. The spherical surfaces were finished by grinding to close tolerances followed by lapping. Each platen was designed and built so that the center of the spherical surface is in the surface of the platen which comes in contact with the specimen and at the same time is at the center of the same surface. Checking of this location of the sphere centers has indicated errors less than 0.002 in. in the case of this particular apparatus.

The fixtures as shown in Fig. 2, were designed primarily for use in a vertical testing machine; consequently it was necessary in the case of the upper fixture, to provide some means for holding the spherically-seated platen in place when there was no specimen in the machine. This has been accomplished by use of ball-bearing gimbal rings attached to the support *a* and connected to the spherically-seated platen *d* by means of four bolts which, in turn, are provided with spherically-seated heads and nuts. In order to comply with the geometry involved it was necessary to have the centers of the spherical surfaces of these parts come at definite locations. The centers of the spherical seats for the heads of these bolts are located in the same plane as the center of the main spherical seat of the fixture, and the centers of the spherical seats of the nuts on the bolts are in the same plane as the axes of the gimbal rings. The four connecting bolts restrict to a considerable extent the horizontal rotation of the platen of the upper fixture but the platen of the lower fixture is quite free to rotate in a horizontal plane. A soft metal bumper ring *f*, has been provided to prevent damage when testing column specimens to failure and to limit the maximum rotation of the platen in any vertical plane. The spherically-seated platens can be readily changed to fixed platens by the insertion of four distance pieces *e*. These distance pieces could be made adjustable so as to correct readily for errors in parallelism of the platens. Suitable eye-bolts are provided for use in handling the fixtures.

The first set of these compression fixtures was designed for a maximum capacity of 300,000 lb. and has been loaded to that capacity in a number of instances. The surface of each platen coming in contact with the test specimens is 24 in. square. The radius of the spherical bearing surfaces of each platen is 10 in. and there is provision for a maximum rotation of the platens of ±5 deg. from the horizontal. The height of each platen, as indicated in Fig. 2, is, of course, dependent to some extent upon the particular testing machine in which the platens are to be used—in this case a 300,000-lb. capacity Amsler hydraulic testing machine (Type 150 SZBDA 233).[6] The various load-carrying parts of the fixtures were designed so that the bending stresses occurring in them would be as small and as uniformly distributed as possible, under the conditions imposed. This was done for the purpose of minimizing the deformations in the fixtures during use. The weight of the top or upper fixture complete was approximately 1625 lb. and of the bottom fixture 1485 lb.

The adaptation of these heads to the Amsler type hydraulic testing machine was readily accomplished and the need for an extra pump eliminated. It was found that the heads could be readily operated with some of the oil delivered by the pump provided with the testing machine. The use of this amount of oil decreased slightly the maximum rate of load application of the testing machine, but since compression tests are generally carried out at relatively low speeds, this was of no practical consequence. It is believed that fixtures of this type could be readily applied to other commercial types of hydraulic testing machines. In the case of screw-power type testing machines it would, of course, be necessary to provide a suitable oil supply for the fixtures.

In working out the details of this testing apparatus, consideration has been given to a number of minor items which facilitate its use. A simple means for fixing the heads against rotation has already been mentioned. An additional feature involves the provision of clips that can be attached to the center of each of the edges of the platens. These clips are provided with adjusting screws which, together with suitable extensions, can be used for accurately locating and maintaining the position of the ends of the specimens. Small brackets are provided at each of the corners of the platens for deformation measuring apparatus. This deformation measuring apparatus is adjustable for various lengths of specimens and affords a means of measuring the relative movement of the platens during use.

In addition to the eye-bolts for handling the heads, unions have been provided in the hydraulic piping at locations where they can be conveniently disassembled when the fixtures are removed from the testing machine, and also where they will not interfere with the other uses of the machine. Experience to date has indicated that the heads function properly when using the same oil as normally used in the testing machine. It is, of course, essential that the oil be clean and free from air.

A number of different procedures may be used to check the functioning of compression testing fixtures of this type. Two schemes that have been applied to this particular set of fixtures will be described and the results obtained indicated.

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[6] "300,000-lb. Universal Testing Machine," *The American Machinist*, European Edition, July 19, 1930.

The centers of the radii of the spherical surfaces of the platens can be made to coincide by bringing the specimen surfaces together and centering. In this position the fixtures can be loaded as a complete sphere up to the maximum capacity (150 tons), and their resistance to rotation in any direction measured. This resistance was conveniently determined by means of a spring balance and a lever arm of known length. The results obtained are shown in Fig. 3. Slight errors in centering the fixtures are reflected in the differences observed in rotating the fixtures in the two planes indicated. The maximum value of the resisting moment measured was about 1800 in-lb. for a load of 175,000 lb.

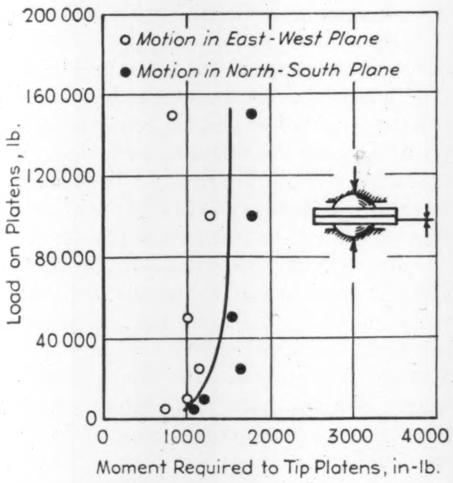

FIG. 3.—Resistance to Tipping of the Fixtures When Functioning as a Complete Sphere Under Load.

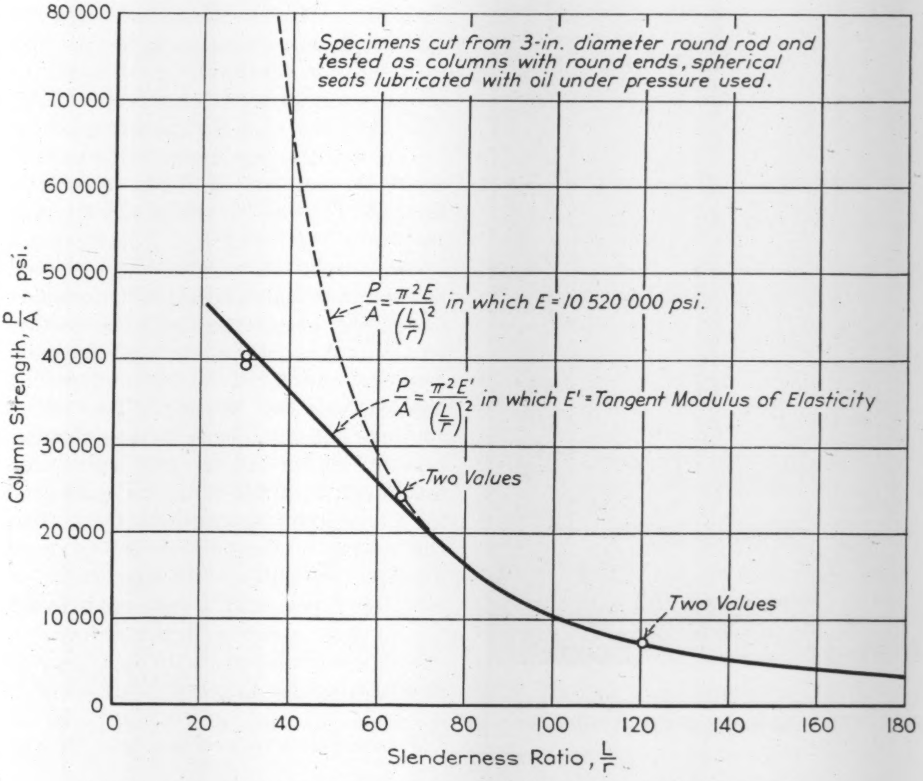

FIG. 4.—Round-End Column Test Results Obtained Using the Apparatus.

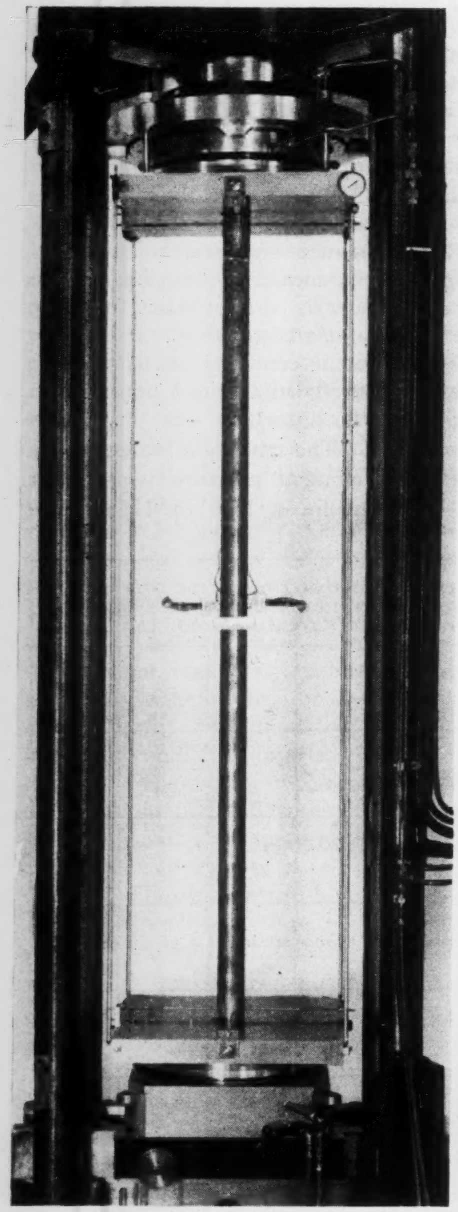

FIG. 5.—Aluminum Alloy Column with Round-End Conditions, During Testing.

This corresponds to 900 in-lb. for each fixture.

It may be pointed out that this checking procedure is a severe one since relatively small errors in centering the fixtures cause considerable resistance to rotation. In fact it appeared reasonable to expect that there might be appreciably less resistance to rotation when testing specimens with the apparatus, particularly in the case of columns. Furthermore, a comparison of the calculated strengths of some given columns with test results obtained with this apparatus would give another check on how closely actual round-end conditions could be attained.

Some column tests have been made using the fixtures and the results are shown in Fig. 4. One of the specimens is shown during test in Fig. 5. In order that the column strengths could be calculated closely, it was necessary first to determine the modulus of elasticity of the material used for the specimens. This involved careful calibration of the testing machine used, in accordance with A.S.T.M. Standard Methods of Verification of Testing Machines (E 4 – 36)[7]; and of the Martens Mirror Extensometer used, in accordance with the proposed methods of calibrating extensometers now under consideration by the Section on Calibration of Testing Machines and Apparatus of A.S.T.M. Committee E-1 on Methods of Testing. Two short compression specimens, having slenderness ratios of about 16, were then machined from the same material used for the column specimens. Data from the testing of these specimens gave an average modulus of elasticity of 10,520,000 psi., and a relation between the average stress and the reduced or effective modulus of elasticity for use in determining the Engesser column curve.

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[7] 1939 Book of A.S.T.M. Standards, Parts I, II and III.

The final calculated column strength values are given by the curve shown in Fig. 4. The column formulas used were the well-known Euler[8] formula:

$$\frac{P}{A} = \frac{\pi^2 E}{\left(\frac{L}{r}\right)^2}$$

and the Engesser[9] formula:

$$\frac{P}{A} = \frac{\pi^2 E'}{\left(\frac{L}{r}\right)^2}$$

where

P/A = average stress at failure in pounds per square inch,

E = modulus of elasticity in compression in pounds per square inch,

E' = reduced or effective modulus of elasticity in compression in pounds per square inch, and

L/r = slenderness ratio of the column specimen.

The column test results are plotted as individual points in Fig. 4 and also given in Table I together with the computed values. It may be noted that these test data check quite closely the computed column strengths.

The long specimens, which failed at average stresses in the elastic range, had strengths about 2 per cent greater than the values computed by means of Euler's formula. This difference represents a fixity coefficient, K, equal to about 0.99 as compared to unity for theoretically round-end specimens and 0.50 for fixed ends.

The test results for the column specimens of intermediate length lie between the curves of the Euler and the Engesser formulas and agree very well with the Euler curve. It is shown in Table I that they are about 4.4 per cent greater than the values computed from the Engesser formula.

The test results for the short column specimens are from about 3 to 5 per cent less than the values computed from the Engesser formula. In view of the scatter of test data usually encountered in this range of slenderness ratios, this variation is not considered large.

TABLE I.—COMPARISON OF COMPUTED COLUMN STRENGTHS WITH TEST RESULTS.

3-in. diameter round rod, aluminum alloy 17S-T

Specimen	Length of Specimen, in.	Slenderness Ratio	Computed Column Strength, psi.	Test Result		Ratio of Test to Computed Values
				Ultimate Load, lb.	Column Strength, psi.	
No. 90A....	90.12	120.2	7 170	51 770	7 290	1.017
No. 90B....	90.12	120.2	7 170	52 120	7 340	1.024
No. 49A....	49.06	65.4	23 000	171 000	24 070	1.047
No. 49B....	49.06	65.4	23 000	170 300	23 980	1.043
No. 23A....	22.69	30.3	41 500	286 600	40 350	0.972
No. 23B....	22.69	30.3	41 500	279 200	39 310	0.947

An analytical study of the effects of restraining moment on ultimate column load, indicated that the smaller the eccentricity of loading the larger the degree of fixation produced by a given small amount of restraining moment. Experience shows that it is practically impossible to achieve zero eccentricity. If zero eccentricity were attained, the degree of fixation at the ends of a column caused by a very small restraining moment would be relatively large. Computations indicated that an eccentricity of loading of 0.009 in., coupled with the resisting moment measured in the friction tests described above, would explain the degree of fixation indicated in the tests of the long specimens. Since the eccentricities in these tests were probably less than 0.009 in., the restraining moment necessary to account for the indicated degree of fixation would be even smaller than that measured in the friction tests; and it is believed that such was the case.

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[8] S. Timoshenko, "Theory of Elastic Stability," pp. 64–69, Engineering Societies Monographs, McGraw-Hill Book Co., New York, N. Y. (1936).

[9] F. Engesser, *Zeitschrift* für Architektur und Ingenieurwesen, p. 455 (1889). See also Considère, Congrès International des procédés de construction, Paris, Vol. 3, p. 371 (1889); and E. Elwitz, ***Forschungsarbeiten***, No. 236 (1921).

As a result of these studies it is concluded that these compression testing fixtures represent a definite improvement over previously available equipment used for similar purposes. While the apparatus is not entirely free from frictional restraint, the friction in it is of a satisfactory low order of magnitude. The design of the apparatus is such that high load capacity is readily obtained; the desired freedom of rotation is provided; distortion of the component parts is minimized; and the apparatus can be used effectively in obtaining either uniform axial loads or loads with a definite eccentricity. The apparatus also meets other less important requirements concerning adaptability to existing testing machines, manipulation, initial cost and maintenance.

Acknowledgment:

In closing the author wishes to acknowledge the assistance of Marshall Holt of the Aluminum Research Laboratories in the carrying out of the tests discussed herein.

ROUND-TABLE DISCUSSION ON THE SOLVENT ACTION OF WATER VAPOR AT HIGH TEMPERATURE AND PRESSURE

GENERAL INTRODUCTION

BY MAX HECHT [1]

Committee D-19 on Water for Industrial Uses, of the American Society for Testing Materials, sponsored a Symposium on Problems and Practice in Determining Steam Purity by Conductivity Methods (**52**)[2] in June, 1941, at Chicago. The papers that were presented reported the current methods for sampling and testing steam condensate, and from that survey there was secured up-to-the-minute information of the quantitative data on carry-over that contribute to deposits in superheaters and turbines. Thus, it is both logical and appropriate that we now discuss the factors, both physical and chemical, that contribute to the formation of deposits; mechanical means that minimize these harmful effects; and the modes of chemical treatment in current use to prevent deposition of salts beyond the steam take-off of the boiler drum.

A twofold objective may be secured from this discussion: 1. The immediate one is to assemble information of current practices found effective for reducing the loss in capacity and efficiency of turbines, for minimizing equipment outages, and to distribute the pooled information to the industry. 2. An objective of greater scope and duration concerns itself with the assembly of all of the critical information that may serve as the basis for a well-balanced research program. This material may include a thorough analysis of all of the concepts previously published, as well as those that may be developed at this discussion.

Plant operators recognized the deposit difficulties when the boiler outputs were increased coincidental with the use of higher pressures and increase in superheater temperatures. Boiler and other equipment manufacturers have attempted to meet these conditions of carry-over by installing steam washing and separating devices in the boiler drum, prior to the steam entering the superheaters. Modes of chemical treatment have been developed to alter the composition of boiler water salts, so that any residual deposits found in the equipment beyond the boiler could be more easily removed by chemical washing or mechanical means.

The general problem of elimination of deposits in superheaters and turbines should be capable of solution, even though the physical and chemical aspects now appear to be complex. Are the deposits formed solely by the mechanical carry-over of boiler water salts in the steam, are they the result of vaporization according to the theory of Spillner (**43**), or are they a combination of these two causes? Although the problem has been with the engineering men for a considerable number of years, it was approached by Straub (**50**) whose suggestions on the use of sodium sulfate have been carried out in certain plants and have resulted in reduction in deposit formation. In recent months Buckland (**4**) has contributed a highly significant and informative paper in which he discusses his findings as the result of his observations of deposits in many turbines. Buckland discusses the subject under the topics: (1) Prevalence of deposits, (2) appearance and location in the turbine, (3) resultant reduction in efficiency, (4) the means of their removal, and (5) general causes and prevention.

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[1] Advisor, Power Stations Chemistry, Pittsburgh, Pa.

[2] The boldface numbers in parentheses refer to the reports and papers appearing in the bibliography appended to this discussion, see p. 1020.

Members of the Power Stations Chemistry Subcommittee of the Edison Electric Institute have reported on a preliminary survey on Turbine Blade Deposits (**54**).

Several papers have been published in recent years, covering the testing of steam purity, and in each of the papers steam purity of exceptionally high quality was reported. Must it be concluded from the results reported that the deposits observed are the result only of the residual carry-over not removed by the washing and separating devices?

Baker (**2**) finds steam purity averaging 0.29 micromhos in boilers operating at 1400 psi. at the Springdale Station of the West Penn Power Co.; nevertheless deposits are found on the turbines after some eight months of continuous operation (privately communicated by L. E. Hankison, West Penn Power Co.)

Schwartz, Gurney, and Crossan (**39**) presented valuable results in their series of papers on testing steam purity. They find by gravimetric analysis values for total solids of from 0.25 to 0.45 ppm. for steam.

Cassidy (**5**) stated:

"The actual maximum steam velocity in modern high-capacity boiler drums may generally exceed the critical so that any moisture picked up from the surface of the water will be carried by the steam except at reduced outputs. Water-separating devices such as baffles which separate the water by change in direction and surface contact are depended upon to deliver comparatively dry steam to the superheater. Effective steam separators are available and are guaranteed to deliver steam with not more than 0.25 per cent moisture. Modern high-capacity boilers equipped with steam washers and separators have been operated at full load with less than 1 ppm. solids in the steam at boiler-water concentrations up to 1280 ppm. The use of the combination steam separator and washer is of great advantage in reducing the deposit of silica on turbine blading and increasing the continuity of efficient service from the turbine."

Lacking a complete exposition of all of the factors that contribute to the formation of deposits, there may be instances where a partial remedy is secured by steam washing in the drum, by the use of separators, or a modification in the boiler water circulation design of the boiler. Steam washing of the turbine during low-speed operation or the use of chemical washes may supply partial relief in other instances. Modification of the chemical treatment of the boiler water may in still other instances assist in a partial solution of the problem. Until there has been made available the fundamentals as to the role of silica and silicates in the formation of the deposits and a general acceptable understanding of the solubilities of the complex salts that are found in boiler waters, the absolute prevention of deposits in superheaters and turbines may have to be deferred.

Is there significant relationship between the formation of silicate rocks and the formation of silicate compounds in turbine blade deposits? Has the geophysicist any data to affirm or disprove the vaporization theory advanced by Spillner (**43**)? Is it possible for him to develop the data secured by him at the unusually high pressures, so as to secure evidence of what occurs in steam generated at pressures up to 1800 psi., and at temperatures up to 950 F.?

A hint may be found in the paper by Morey and Ingerson (**28**), investigators at the Geophysical Laboratory, Carnegie Institution of Washington, Washington, D. C. The senior author, G. W. Morey, both because of his investigations on silicate rocks and researches on glass, may be able to blaze the trail on this subject of superheater and turbine blade deposits. His findings should benefit the industry, the operators, boiler and turbine manufacturers, and the responsible consultants and chemists charged with the function of maintaining continuous and efficient operation of power equipment, and his paper introduces this Discussion on the Solvent Action of Water Vapor at High Temperature and Pressure.

SOLUBILITY OF SOLIDS IN WATER VAPOR

By George W. Morey[1]

The interest of the Geophysical Laboratory of the Carnegie Institution of Washington in the carry-over of silica and other nonvolatile substances in high-pressure steam arises from the fact that our first interest is the application of the methods of chemistry and physics to the solution of the questions raised by geologists in their study of processes of rock formation and mineral deposition. These studies by the geologists indicate that transport of nonvolatile material by a vapor under high pressure may play an important part, and the dominant constituent of this vapor is water. In other words, we have in nature much evidence which has been interpreted as indicating the transport of nonvolatile material by steam at high pressure and temperature.

Most ore deposits of economic value are associated with igneous intrusions, but the main body of the batholith or laccolith is usually barren. The deposits have been formed by ore-bearing solutions emanating from the hot magma, and depositing their load in veins and crevices at some distance from the main intrusive body. The water vapor at high temperatures and high pressures exerts a solvent action on comparatively nonvolatile materials, and the highly charged vapors penetrate pores in the country rock, especially those of sedimentary origin such as limestones, shales, and sandstones. Many tin, tungsten, copper, molybdenum and mercury, ores, are examples of high-temperature deposits of this type.

That stage in the process of magmatic differentiation which is known as pneumatolytic is one in which the part played by water at temperatures far above its critical temperature and under high pressure is of special importance. In the early stages of pneumatolytic action, in which the residual magma represents the mother liquor remaining after the crystallization of the plagioclase feldspars and more basic minerals, the chief minerals separating are quartz and orthoclase. Each of these minerals is characterized by high melting point, and orthoclase, in the laboratory, is impossible to obtain crystalline without the aid of mineralizers. Nevertheless, pegmatite deposits known to be laid down at temperatures not very far from a red heat contain crystals of orthoclase and quartz, and some such are of enormous size. Their formation can be explained only by their being in solution in a highly mobile phase, the major constituent of which is water, together with accessory constituents such as the halogen acids, phosphates, carbonates, and boric acid. The part played by the solvent action of water at temperatures above its critical temperature is a dominant one in the late stages of magmatic differentiation, and our major interest for the past twenty years has been the laboratory study of systems which we hope will lead to a clearer understanding of this process.

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[1] Geophysical Laboratory, Carnegie Institution of Washington, Washington, D. C.

That experimental work has unfortunately been temporarily stopped by the necessity of playing our part in our country's war effort. We have, however, obtained many results which may be significant in their application to the broad problem, and some of them may have a direct bearing on the carry-over in high-pressure steam.

Before considering water as a magmatic constituent, it may be well to consider the properties of water itself. Figure 1 shows the steam dome, that is, shows the density of water in the coexisting steam and liquid phases. For our purpose, it is noteworthy for the fact that while the density of water decreases, that of steam increases, and the increase in density of steam is very rapid as the critical temperature is approached. At and just below the critical temperature, the steam dome is almost flat. From the principle of continuity, it follows that if we heat water under a pressure always greater than its vapor pressure, it will pass without discontinuity in properties from a condition in which it is plainly recognizable as liquid water to a condition at temperatures above its critical temperature where it must be regarded as a gas. There is no discontinuous change at the critical temperature of water, and there is no reason to expect a change in the solvent power of water at the critical temperature, when the liquid and vapor phases merge continuously into each other.

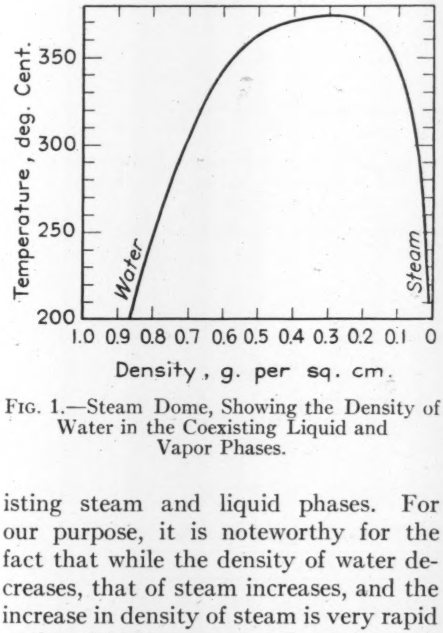

FIG. 1.—Steam Dome, Showing the Density of Water in the Coexisting Liquid and Vapor Phases.

The critical temperature of water is a characteristic of pure water only. When a binary system consisting of water and a soluble compound such as potassium disilicate is considered, the critical temperature of water is without significance. A solution of salt in water raises the boiling point, that is, decreases the vapor pressure, and if the salt is sufficiently soluble the vapor pressure of the saturated solution at the critical temperature of water is far less than the vapor pressure of pure water. Likewise, the critical temperature of water is altered by dissolved salt, and usually we may speak of the critical or plait-point curve as joining the critical point of water with that of the dissolved substance. When the solute is present in sufficient quantity, the vapor-pressure curve of the three-phase equilibrium solid + liquid + vapor, does not intersect the plait-point curve, and we have conditions which are represented by the binary system, water-potassium disilicate, to be considered later.

Before considering this binary system, it would be well to discuss in general terms the phase equilibrium relationships in systems the components of which differ greatly in volatility. Figure 2 gives three projections of the solid model representing the variations in pressure, temperature, and composition of the three-phase equilibrium solid + liquid + vapor for a binary system $A + B$, in which A is much more volatile than B. The three projections are (1) that on the T–X plane, which is the ordinary solubility curve, continuous from the eutectic or cryohydrate to the melting points of each of the components; (2) that on the P–T plane, which represents the change of pressure with temperature of the three-phase equilibrium solid + liquid + vapor; and (3) the P–X plane, which represents the variation of pressure with composition in the same equilibrium.

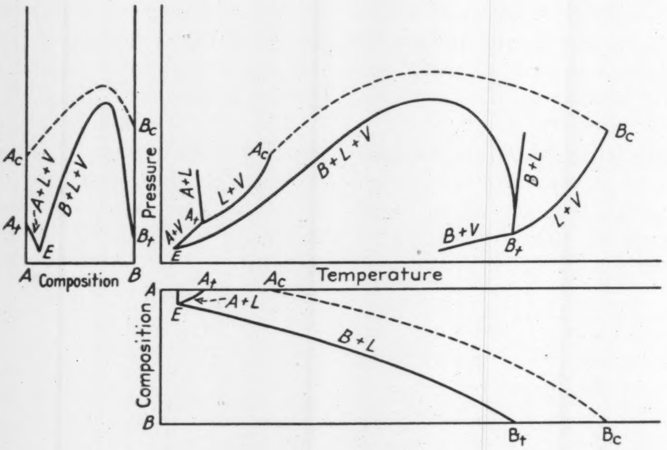
A_c
A_t
A+L+V
B+L+V
E
B_c
B_t
A
Composition
B
Pressure
A+L
A_t
A+V
L+V
A_c
B+L+V
B+L
B+V
B_t
L+V
B_c
E
Temperature
A
A_t
A_c
A+L
E
B+L
Composition
B
B_t
B_c

FIG. 2.—Three Projections of a Solid Model Representing Variations in Pressure, Temperature, and Composition of the Three-Phase Equilibrium Solid + Liquid + Vapor, for a Binary System $A + B$, in Which A is Much More Volatile than B.

The critical, or plait-point, curve (dashed) is not intersected by the solubility curve (solid).

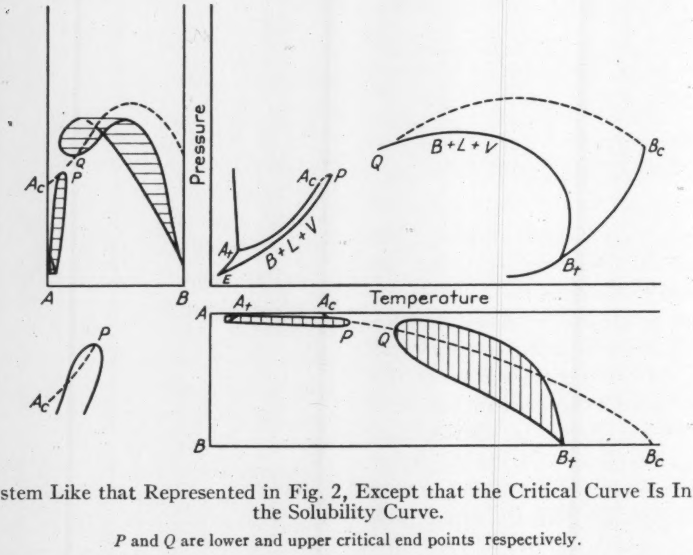
Q
P
A_c
A
B
Pressure
A_c
P
A_t
B+L+V
E
Q
B+L+V
B_c
B_t
Temperature
A
A_t
A_c
P
Q
P
A_c
B
B_t
B_c

FIG. 3.—System Like that Represented in Fig. 2, Except that the Critical Curve Is Intersected by the Solubility Curve.

P and Q are lower and upper critical end points respectively.

On the pressure-temperature curve of this three-phase equilibrium, the pressure rises to a maximum and then falls.

This is necessarily true because this curve terminates at the melting point of *B*, and the pressure at that point is the triple-point pressure of *B*. Similarly, the critical temperatures of the two components are joined by a critical or plait-point curve, and in the case represented by Fig. 2 this plait-point curve is not intersected by the solubility curve.

Figure 3 shows the relationships in the case that the critical curve is intersected by the solubility curve; further assumptions are that no compounds are formed and that no unmixing takes place. When the critical curve is intersected at one point by the solubility curve, it must necessarily be intersected at a second point. As the temperature is increased from the eutectic to the first critical end point, the compositions of the two phases approach each other; and at the critical end point they become identical. Between the two critical end points there is a region in which liquid solution or melt cannot be obtained no matter what the pressure. The only possible phases are solid and vapor; the system is divariant, and composition of the vapor in equilibrium with solid is a function of both pressure and temperature. At the second critical end point, a liquid is again formed of composition identical with the vapor, and at higher temperatures there is again a three-phase curve ending, as in the preceding case, in the melting point of component *B*.

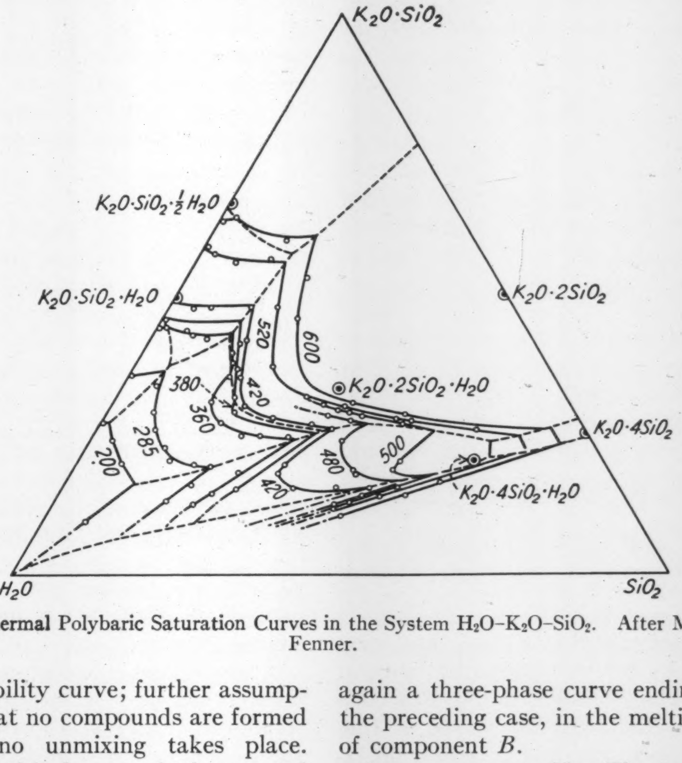
$K_2O \cdot SiO_2$
$K_2O \cdot SiO_2 \cdot \frac{1}{2}H_2O$
$K_2O \cdot SiO_2 \cdot H_2O$
$K_2O \cdot 2SiO_2$
$K_2O \cdot 2SiO_2 \cdot H_2O$
$K_2O \cdot 4SiO_2$
$K_2O \cdot 4SiO_2 \cdot H_2O$
H_2O
SiO_2
600
520
420
380
360
285
200
480
500
420

FIG. 4.—Isothermal Polybaric Saturation Curves in the System H_2O–K_2O–SiO_2. After Morey and Fenner.

Let us now consider Fig. 4, the ternary system water–potassium metasilicate–silica (**27**).[2] In this ternary system we have binary systems in which the critical curve is not intersected by the solubility curve, and also binary systems in which the critical curve is intersected by the solubility curve. The diagram as a whole consists of a continuous series of isothermal polybaric saturation curves from 200 up to 600 C. The sequence of these saturation curves is continuous, and the critical temperature of water is without influence.

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[2] The boldface numbers in parentheses refer to the reports and papers appearing in the bibliography appended to this discussion see p. 1020.

The binary system H_2O–$K_2O \cdot SiO_2$ is continuous up to the melting point of potassium metasilicate, and the maximum pressure on this curve is comparatively low. The relationships in the binary system H_2O–$K_2Si_2O_5$ are complicated by the formation of a hydrate, $K_2Si_2O_5 \cdot H_2O$, but except for this complication, the solubility curve is continuous, and again the critical temperature of water is without influence.

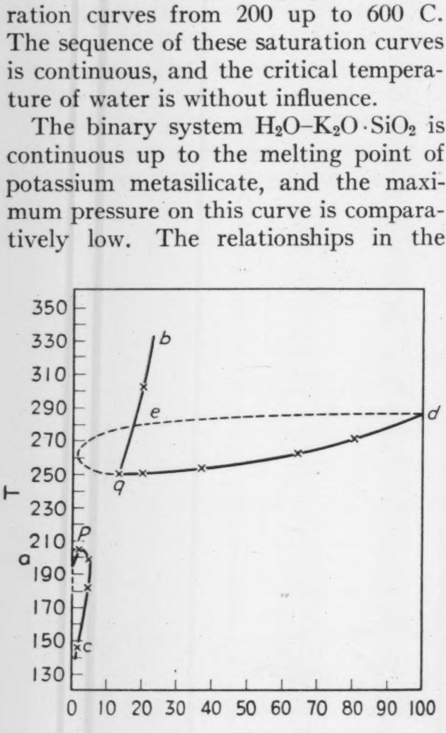

FIG. 5.—*T–X* Curve for the Binary System Ether–Anthraquinone. After Smits. Compare Fig. 6.

At lower temperatures, the relationship between water and potassium disilicate changes from that of congruent solubility to that of incongruent solubility, a type of relationship which may be of importance in connection with the carry-over in steam turbines. In the region in which anhydrous potassium disilicate is solid phase, and in the upper part of the region in which the potassium disilicate monohydrate is solid phase, these compounds crystallize out of a solution in which the ratio of K_2O to SiO_2 is that of the disilicate. That is, the compound is not decomposed by water. When the temperature is lowered to below 280 C., this is no longer true. The disilicate becomes decomposed by water and crystallizes only from solutions which contain an excess of potassium oxide. If a solution containing potash and silica in the disilicate ratio is cooled and crystallized below 280 C., the compound separating is not potassium disilicate but potassium tetrasilicate, and below this temperature potassium disilicate is incongruently soluble in water.

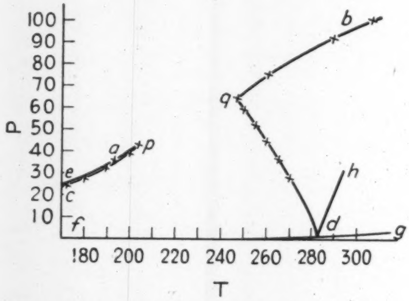

FIG. 6.—*P–T* Curve for the Binary System Ether–Anthraquinone. After Smits.

A larger number of compounds become decomposed by, and are incongruently soluble in, water at elevated temperatures. Potassium tetrasilicate becomes decomposed by water at temperatures below 360 C. with quartz becoming the stable phase. When incongruent solubility is accompanied by a volatility of one or the other components, the probability of carry-over is greatly increased.

The binary system H_2O–SiO_2 is an example of one in which the plait-point curve is intersected by the solubility curve, but a better example is the classical system ether–anthraquinone, worked out by Smits (**40**). Figure 5 shows the *T–X* curve for this equilibrium, and Fig. 6 shows the *P–T* curve. The real solubility of anthraquinone in the vapor phase is clearly evident in this case.

In attempting to extend the work with potassium silicates to the sodium silicates,[3] it was found that the lower solubilities resulted in greatly increased vapor pressures and, consequently, increased difficulties in the experimental technique. Experiments at comparatively low temperatures have shown that the solubility of sodium metasilicate is strongly retrograde. For example, at 100 C. the saturated solution of sodium metasilicate in water contains about 50 per cent of the anhydrous compound, but at 350 C. the solubility is measured only in tenths of a per cent. We also have found that at 350 C. sodium disilicate is rather more soluble than sodium metasilicate, and that the greatest concentration of anhydrous material is at the boundary between sodium disilicate and quartz. It is also important to notice that at this temperature the boundary between disilicate and quartz lies almost at the disilicate ratio, and it is probable that at lower temperatures sodium disilicate becomes decomposed by water. It has not been established, but it is inferred from the preceding results that the region of critical end points extends over a considerable portion of the ternary system H_2O–Na_2O–SiO_2. In other words, it is probable that saturated solutions of sodium silicate in water, over a considerable range of $Na_2O:SiO_2$ ratio, show critical phenomena.

Because of the high pressures involved, it was necessary to devise a new method of experimental procedure for the study of the system at high temperatures. The plan of experimentation finally adopted was to heat small quantities of mixtures of varying $Na_2O:SiO_2$ ratio, in the form of homogeneous glasses, in steam at high pressure long enough for equilibrium to be reached; then to cool the charge by dropping it out of the heated zone of the furnace, thus freezing the equilibrium. The experimental set-up consists of a stainless steel bomb 6 in. in external diameter, with a 3-in. central cavity, and provided with appropriate means of heating, of temperature measurement, and with an electrical device for dropping the charge of sodium silicate glass, wrapped in platinum foil, to the bottom of the furnace at the termination of the experimental period. In the central cavity is an electrically heated, platinum wound, "Sillimanite" tube $\frac{1}{2}$ in. in internal diameter, which can be independently heated to temperatures up to 1300 C. The bomb walls are maintained at a constant temperature higher than the critical temperature of water to preclude any possibility of condensation. The pre-heated water vapor is introduced through the side wall of the bomb by means of an injector pump, the operation of which is controlled by an electrical device which maintains the pressure constant. The apparatus, however, is so tight that after the pressure has once been built up, it is maintained by the pump operating less than one minute out of 24 hr. By this means, we have been able to study the equilibrium relationships in the system water and sodium silicates at high temperatures and pressures. Three groups of experiments, each of which, in a different manner, is pertinent to the solubility of solids in a vapor are described below.

The first of these groups of experiments has to do with the determination of the upper portion of the solubility or T–X curve. Let us confine our discussion chiefly to sodium disilicate, with incidental references to systems containing larger or smaller proportions of silica. The melting point of sodium disilicate is 874 C. When quenching experiments are carried out in steam, the water dissolved in the molten silicate lowers the freezing point of the silicate in an amount determined by the pressure of the water, and the freezing point or solubility curve so determined is shown in Fig. 7. This is the P–T curve, and it shows that 3000 psi. pressure of steam is sufficient to lower the freezing point from 874 to 708 C. At the binary eutectic between sodium disilicate and quartz, the temperature lowering is 156 C., and it appears that the silica content at this eutectic has changed only a little.

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[3] A preliminary paper on the system H_2O-Na_2O-SiO_2 was presented by Morey and Ingerson (**29**). Much of the work subsequently referred to is from unpublished studies by them.

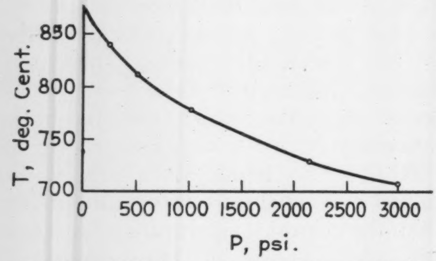

FIG. 7.—*P*–*T* Curve for the Binary System $Na_2O \cdot 2SiO_2$–H_2O, up to 3000 psi.

Experiments of this type have been carried out for the entire range of sodium silicates up to sodium metasilicate, and also for the system sodium disilicate-albite and sodium disilicate-nephelite, as well as in many mixtures in the system Na_2O–CaO–SiO_2. The details of these experiments will be published at another time, but some phenomena noticed in the course of these experiments are of great interest to us in this problem.

In all this work there is constantly being obtained evidence of transport of ordinarily nonvolatile materials by high pressure steam, but the most striking examples were obtained with a bomb in which there was an undue amount of convection. One of the major difficulties in this experimental work is that it is necessary to have a region of constant temperature in that portion of the ½-in. "Sillimanite" tube in which the quench and thermocouple are located, and since convective currents in the steam at several-thousand-pounds pressure carry quantities of heat, the avoidance of convective currents is imperative. Numerous experimental arrangements were tried, and one of the bombs in which convection was not entirely eliminated was used in some experiments at about 1000 C. and 3000 psi. pressure. When the thermocouple, in its protective tube, was removed at the end of the experiment, it was noticed that there were many hair-like crystals on the thermocouple leads and on the bottom of the refractory tube. Above the needles in a zone which had been between 800 and 900 C., there was a brownish coating, then a platy white coating, and finally a thicker, irregular, lumpy white layer. Microscopical examination showed the needles to be sillimanite; the brownish coating, rutile; the plates, corundum; the lower part of the uppermost zone, quartz and amorphous silica. Other deposits in the bomb were mostly amorphous silica which was deposited in quantity near the top, and between baffle plates where the steam first cooled by the colder shell after passing upward through the hot spot in convectional circulation.

This is, I believe, the first time that sillimanite has been obtained artificially, and it is probable that the conditions under which it was formed in these experiments resembled those in which it was formed by metamorphism in nature. The anhydrous materials such as silica, alumina, and titania had their source in the refractories from which they were dissolved by the highly compressed water vapor, by which they were transported from one region of the bomb to another. This transport, of course, took place under conditions of much higher temperature and pressure than are ever met with even in superheaters, but that the same process can and does take place at lower temperatures and pressures is not to be doubted.

The third and final group of experiments which I wish to mention are a continuation of our efforts to realize the upper portion of the solubility curve of sodium disilicate in water. Reference to Fig. 7 will show that the *P–T* curve is concave upward; that is, a given increment of pressure is producing an increasingly smaller depression of the freezing point. We had expected that this system would resemble the system water-potassium disilicate, and that the *P–T* curve would show signs of approaching a maximum in pressure, but that proves not to be the case. Then some experiments were made in which the temperature was lowered to just above the critical temperature of water at 3000 psi. pressure. The crystalline sodium disilicate showed no sign of melting. The finely powdered grains were not even sintered together. It is evident that the system is one in which critical end points are formed, and it is probable that the lower critical end point differs only slightly from the critical temperature of water. This is in harmony with the solubility experiments at 350 C. previously mentioned.

We then tried to determine the upper critical end point. In these experiments, instead of a quenching charge, we used a tiny platinum crucible. At 500 C. and 8500 psi. pressure, the crystals of sodium disilicate were not melted, but above them and on the sides of the crucible, a gelatinous layer had deposited. At 8700 psi. pressure the disilicate had entirely melted. In the cooled crucible there were found two distinct layers. One of these was a quenched disilicate glass containing water, but still hard and brittle. The upper layer was of gelatinous consistency, containing much more water, and evidently represented a chilled vapor phase.

Numerous similar experiments have been made at temperatures ranging from 375 to 500 C., and at pressures up to 16,500 psi. and with mixtures ranging in composition from sodium metasilicate to the eutectic between sodium disilicate and quartz. These studies are not complete, but they all indicate that under the conditions of experiment the percentage of sodium oxide and silica in the vapor becomes large. Some results, as yet unconfirmed, indicate over 50 per cent by weight of solid material in solution in the vapor.

The experimental work which has been discussed was planned in the hope of obtaining information on the behavior of systems containing water at temperatures and pressures high enough to be comparable with those produced during igneous differentiation and also in the formation of metamorphic rocks, and they are of especial interest in that for the first time we have experimental results which directly illustrate the rather abstruse theory of critical phenomena in binary systems containing components of widely differing volatility. The conditions of these experiments are more drastic than those encountered in steam boilers and superheaters, but nevertheless the very considerable power of steam under these more drastic conditions to transport ordinarily nonvolatile components will be paralleled by a smaller transport power per unit of steam at the lower pressures. However, when the enormous quantity of water which passes through one of these turbines is considered, it will be evident that the possibility of transport of nonvolatile materials by steam at the pressures in commercial use is capable of producing the deposits which so greatly impair the efficiency of the turbines. Solubility of nonvolatile materials in a compressed, highly heated gas or vapor is a real phenomenon of importance not only to the geologist, but also to the engineer concerned with the efficient operation of modern high-pressure power plants.

GENERAL DISCUSSION—ROUND-TABLE DISCUSSION ON THE SOLVENT ACTION OF WATER VAPOR AT HIGH TEMPERATURE AND PRESSURE

MR. L. DREW BETZ[1] (*presented in written form*).—It has been our experience that at lower boiler pressures the chemical characteristics of the boiler water markedly affect steam purity and turbine blade deposits. The mechanism of such carry-over would correspond to Spillner's "Mechanical Theory" (**43**).[2]

There is a marked difference between the type of carry-over and turbine blade deposits developed at lower boiler pressures and those obtained at high pressures. It is realized that there is no sharp line of demarcation between "low pressure" and "high pressure." However, in this discussion we will employ an arbitrary value of 1000 psi. to mark the dividing between low pressure and high pressure.

It is true that mechanical factors such as basic boiler design, influence of load changes, boiler rating, water level, etc., exert influence on steam purity. However, at low boiler pressures the chemical characteristics of the boiler water are of equal importance. It has been our experience that the limiting chemical factor on the production of pure steam is usually the suspended solids content of the boiler water, in the form of calcium phosphate, calcium carbonate and magnesium hydroxide. Particularly when present with relatively high boiler water alkalinities, these suspended precipitates are the prime source of carry-over due to chemical sources.

Of these suspended precipitates normally present in boiler water, calcium phosphate is the worst offender in the production of impure steam. The effect of calcium carbonate and magnesium hydroxide is less marked than the effect of calcium phosphate.

Our own work, initially in the form of laboratory experiments, and later checked over a period of several years in actual plant operation, has indicated that it is possible to overcome the effect of these suspended precipitates in causing carryover. Proper agents will effect agglomeration and adsorption of these suspended precipitates and will so condition the boiler-water sludge as to minimize if not eliminate the effect of this sludge on steam purity. Of the various sludge conditioners studied for this purpose we have found that reactive organic colloids possess to a marked degree the ability to prevent foam stabilization and carry-over of solids with the steam. This is accomplished by adsorption of the suspended precipitates present in the boiler water and their natural tendency for stabilizing boiler water foam is thereby prevented. The use of an agent thereby as a sludge conditioner performs the dual function of minimizing the effect of these precipitates on entrainment of solids with the steam and, secondly, minimizes their tendency for adherence to the boiler heating surfaces.

Numerous tests under plant conditions have shown it possible to increase boiler water solids concentrations as much as

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[1] Member of Firm, W. H. & L. D. Betz, Philadelphia, Pa.

[2] The boldface numbers in parentheses refer to the reports and papers appearing in the bibliography appended to this discussion, see p. 1020.

100 per cent by the use of such a sludge conditioning agent. The procedure usually employed in such a study has been to record continuously the conductivity of the condensed steam from a boiler employing, for example, a straight inorganic phosphate treatment without organic sludge conditioner. Boiler water concentrations are gradually raised, through reduction in blow down, to the point where carry-over of solids with the steam becomes evident. Boiler-water concentrations are relieved by blow down and the process is repeated. In this manner, for the same operating conditions, the limiting solids concentration of the boiler water can be established with respect to carry-over. Under the same conditions, an organic sludge conditioner is then introduced to the boiler water in the proper quantities and with continuous recording of steam purity, concentrations are again raised to the point where carry-over becomes evident. While subject to considerable variation dependent upon the individual plant conditions at hand, it can be stated as a general rule that if the proper organic agent is chosen for the prevention of carry-over, boiler water solids concentration can usually be increased to a value double that which can be maintained with inorganic treatment alone. In some cases, it has been possible to raise boiler water solids concentration to values in excess of 10,000 ppm. with a lower solids content of the steam than experienced with inorganic treatment alone at boiler water concentrations approximating 3500 ppm. It has also been possible to maintain boiler water solids at such a high level that the limiting factor governing the solids content of the boiler water was not the purity of the steam but rather the high suspended solids content of the boiler water with the attendant possibility of baking sludge on the heating surfaces.

Studies in which the above results have been obtained have been run at boiler pressures below 1000 psi. In the examination of high-pressure operation where serious turbine blade deposition has resulted, boiler feedwater is almost invariably of such high quality and the boiler water of such low solids content that the known carry-over effect cannot be explained by the presence of the very small quantities of suspended precipitates such as calcium phosphate present in the boiler water. In plant tests at such pressure we have found that the application of sludge conditioning agents to the boiler water yields little or no effect on the solids content of the steam (already as low as 1.0 ppm. solids or less). In view of the proved results secured at low pressures with this same type of treatment, we have previously concluded that carry-over of solids at high boiler pressures is due to different causes than at low pressures and it is not curable by the same means. We think it well, therefore, that the carry-over of boiler water solids be considered as due to separate causes for low-pressure and high-pressure operation, and that the mechanism of such carry-over and means employed for its prevention be considered in the light of the operating pressure under consideration. While internal chemical treatment may prove to be the complete solution to a carry-over problem at the lower pressures, it is evident that the application of the same treatment to high-pressure operation is practically useless.

Spillner's theory (**43**) on the solvent action of high-pressure steam appears to offer a plausible explanation for conditions frequently found in operation. The solvent action of steam at high pressures may possibly be due to polymerization of the steam molecule H_2O into a form such as $(H_2O)_n$ which is the form of water and ice. It is known (vapor density measurements) that at 100 C. and one atmospheric pressure the vapor density of steam is too great for the molecular formula H_2O and much too small for H_4O_2. It has been assumed that steam contains a mixture of H_2O with a few H_4O_2 molecules and that equilibrium condition for water vapor, $H_4O_2 \rightleftarrows 2H_2O$, corresponds to 91 per cent of H_2O molecules in the vicinity of 100 C. (**23**). Increased polymerization of the steam molecule at high pressures could possibly result in formation of a molecule similar to that of water and thus explain the solvent action exerted.

If Spillner's theory is accepted, no apparent solution to the problem of carry-over in high-pressure operation is evident other than the maintenance of boiler water solids at as low a level as possible and this level is probably dependent on the purity of an evaporator distillate. Since there is no sharp line of demarcation between high and low pressure it is probable that an intermediate pressure range exists with some characteristics of both high and low-pressure operation.

Despite the fact that silicic acid may not be well defined chemically, it would be of considerable value if the behavior of such compounds were investigated due to the predominance of silica in many turbine blade deposits. It is evident that, together with all other salts, the concentration of silica in the boiler water should be kept as low as possible.

It is usual to employ conductivity as the means for measuring steam purity. It is also usual to accept a factor of 0.5 to 0.6 ppm. solids per micromho of conductivity. However, silica present in condensed steam exhibits a lower conductivity per unit of solids than the other common salts carried over with the steam, and in our experience we have found this value to average approximately 0.8 ppm. silica as SiO_2 per micromho of conductivity.

MR. B. O. BUCKLAND.[3]—The deposits observed during turbine inspections may have a close relation to the phenomena Mr. Morey has described. I have seen tiny crystals of what looked like quartz lodged on the nozzle and bucket surfaces of a turbine and have often wondered how the silica could be deposited in that form from the steam. Perhaps it would be useful to describe these silica deposits, their appearance and physical characteristics, and their location in the turbine.

Silica deposits are found in two forms: the sparkling silvery gray crystals, and the film or glassy form. Generally, the crystals are found in that part of the turbine steam path which lies between 600 and 300 F., and the hard-film type in the region between 300 F. and the exhaust. The boundaries are not definite, for scattered crystals have been seen as high as 750 to 800 F., and the crystal layers as low as 250 F. However, I have never seen the crystals below the dew point in the turbine. The crystal type occurs in quantities ranging from only a few scattered crystals per blade to layers completely covering the surface and sometimes to layers $\frac{1}{32}$ in. thick or more. Thicker deposits than $\frac{1}{32}$ in. are found, of course, but these heavy layers usually contain considerable amounts of other substances, principally sodium oxide. The scattered crystals can readily be detected by the sparkle which results when a flashlight is shone tangent to the blade surface. I should estimate that they are about 0.01 in. across.

The film type, which is found from 300 F. down through the wet region and all the way to the last stage of a condensing turbine, does not seem to be accompanied by any appreciable amount of sodium oxide, but the amount of iron oxide present increases rapidly as the exhaust is approached. The iron oxide content is probably responsible for the rusty hue which this deposit takes on in the latter stages. Rather surprising amounts of organic matter are also found in this type. C. Dantsizen has suggested that the organic content could be oily substances distilled over with the steam from the boiler and condensate system.

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[3] Turbine Engineering Dept., Mechanical Research Section, General Electric Co., Schenectady, N. Y.

A characteristic of all deposit seems to be its persistence in clinging thickest at the insides of the fluid turns, that is, on the convex surfaces of the blades and at other points of rapidly falling pressure. Often the concave surfaces are entirely clean while $\frac{1}{16}$ in. or more of deposit will exist in the convex surface of the nozzle throat. Where the gray crystal layer of silica occurs by itself, however, this characteristic is less marked. The sparkling layers also have another difference in the manner of their occurrence, namely, they are found on the shell walls, the outside of the shroud bands, and on other surfaces not in the direct steam path. The thick layers which contain more soluble matter, however, occur only in the main steam path.

The glassy deposits with high iron oxide content also seem to occur in more uniform layers or films than the deposit with soluble material. They occur predominantly, however, on the convex surfaces of the blades. I would rather suppose the deposit occurs on the convex surfaces due to the fall in steam pressure than that it is due to a fall in temperature or to the thicker boundary layers existing on these surfaces, for two reasons: first, the fall of temperature in the blade surface is probably small; and, second, ridges of deposit are found in thin boundary layer regions on partition noses at points of rapid pressure drop where the stream divides in flowing around the partitions.

Although I have observed silica of the film type in turbines of relatively low initial pressure, I have never seen the gray crystalline type in other than high-pressure turbines, and the number of silica deposits has increased greatly with the increased use of steam conditions of the order of 1200 psi. and 900 F. This fact then might be considered another characteristic of silica, namely, that it occurs frequently with high-pressure and high-temperature steam.

The washing of turbines with condensate removes little if any of the silica deposit. Turbines with silica deposit have been washed with sodium hydroxide and this procedure will probably be developed into a usable technique. However, the losses in capacity and efficiency due to deposit are costly and aggravating and means for preventing the deposit would be much more desirable than means of cleaning. Perhaps until more convenient preventive measures are developed, steps should be taken to reduce by a substantial amount the quantity of silica in the boiler.

MR. C. H. FELLOWS.[4]—Mr. Buckland has indicated very well the distribution of deposits in the turbine barrel. In the high-pressure group of wheels and nozzles will be found, as a rule, deposits that tend to be soluble. In the next group of wheels will be found the more insoluble deposits. In some instances these contain more than 85 per cent silica. This entire subject came up for discussion at a meeting of the Power Division of the American Society of Mechanical Engineers held in Cleveland during the week of June 8. I have been asked to present a summary of the discussion at this meeting.

It was the consensus that at the low-pressure end of the turbine there are very few deposits. Perhaps the average person overlooks what few deposits actually exist in this location, whereas someone examining the blades critically, as Mr. Buckland has done, would discern deposits of a character that the other people overlook.

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[4] Head, Chemical Division, Research Dept., The Detroit Edison Co., Detroit, Mich.

The adverse effect of scale is not only the loss in efficiency, but also its effect on energy distribution resulting from abnormal flow conditions. Another feature that was brought out in the discussion concerned the methods by which turbine blade deposits could be eliminated. Mention was made by several discussers of the use of sodium hydroxide to eliminate scale deposits or blade deposits. Just how sodium hydroxide has been used was not disclosed, but turbine manufacturers were quite emphatic that sodium hydroxide was not desirable. Much to my surprise they considered the possibility of caustic embrittlement in the roots of the blades through use of sodium hydroxide. That is certainly putting caustic embrittlement in a new spot in the system.

Silica deposits were reported where load losses were as high as 30 per cent in six weeks and a consensus is that washing with steam is not recommended unless absolutely necessary. The feeling was that the problem was one for the chemist and it was felt that, as Mr. Buckland stated, the *amount* of material carried by the steam vapor is of less significance than is the *character* of that material. Something must be done if we are to be relieved of this difficulty; the boiler water itself must be so treated that material carried over with the steam as a vapor or as solid particles are capable of passing right on to the turbine without adhering to the blades or nozzles.

MR. R. E. HALL[5] (*presented in written form*).—The veritable epidemic of formation in the turbine of deposits, especially silica, having their origin in boiler water salts has led to the speculation, and to some though not extensive investigation thereof, that the mechanism of transfer of these materials from the boiler water to the steam is by vaporization with the steam; or stated otherwise, by their solubility in the steam.

It is logical to conclude that increased deposition in the turbines results from higher steam temperatures and pressures, higher rates of heat absorption in boiler furnaces and an increasing prevalency of base-load conditions, and is not something now merely more commonly recognized because of the growing demand for greater operating efficiency. While steam pressures are in general below that corresponding to the critical state of water (3206 psi.; 705 F.), steam temperatures both in superheater and turbine are well above. We believe the same can be said of the steam in the boiler, too, if we consider the individual steam bubble at its point of origin, instead of the over-all steam at the steam outlet. High temperature and pressure, the latter especially at high temperature, increase the concentration of mineral salt which the vapor phase, steam, can hold in solution. Since the relationship of solid and vapor that must exist in the region of critical conditions is a problem of tremendous interest to the geophysicists, Mr. Morey (28) has been working in the difficult and unexplored field it represents. His presentation thereof opens to our minds the vista of its application to the problem of turbine deposition. Of particular interest is the fact that the class of salts whose solubility steadily increases with increase in temperature develops entirely different relationships as critical conditions are attained from those which arise with the class of salts whose solubility decreases with increment of temperature and approaches or reaches zero at the critical state of water. Other contributions to this field are not many; a few relating particularly to silica have been listed in the bibliography (**37, 6, 41, 55, 3, 26**).

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[5] Director, Hall Laboratories, Inc., Pittsburgh, Pa.

If boiler-water salts vaporize with the steam, the conclusion is inescapable that steam washers or baffles in the outlet drum cannot be wholly successful; neither can any chemical treatment of the boiler water prevent this effect. Naturally the amount of material carried to the turbine may be strongly increased by mechanical carryover as conventionally defined, but the process of entrainment by vaporization must be given entirely separate consideration. It is apparent therefore that the present intense effort to accomplish mechanical separation of boiler water from outgoing steam must suffer no letdown; but added thereto must be procedure directed to combating the ill effects of transfer in vapor form.

Our purpose in this discussion is to review briefly what facts have been established and relate them in so far as we can to the problem of turbine deposits that confronts us.

CHARACTERISTICS OF TURBINE DEPOSITS

Our attention will be directed to silica, and to the boiler-water salts such as sodium hydroxide, sodium chloride, sulfate, or silicate, which may enter the steam either mechanically or by vaporization, and which in either event must undergo crystallization from solution in the formation of turbine deposits. We shall exclude such materials as calcium phosphate and magnesium silicate characteristic of the boiler sludge, and any incidental metallic oxide, or oil.

Of these, all are soluble but silica, which in reality is a secondary component in that it results from changes occurring in the primary sodium silicate. Because of their solubility, and the moisture in the turbine during the interval intervening between its shutting down and collection of the sample, their relative preponderance in the deposit cannot be accurately known. For instance, in the sample collected during the first minute of washing a turbine, M. D. Baker of the West Penn Power Co.,[6] found 62 per cent of sodium silicate, 22.6 per cent of sodium sulfate; in the second minute, 64.2 and 25.4 per cent, respectively; while in a composite representing the fifth to the tenth minute, the sodium silicate was 23.6 per cent and the sodium sulfate 61.1 per cent. Differences in the dissolving rate of the sodium silicate and sulfate apparently caused these differences in their relative proportions as sampled.

For analyses of individual turbine deposits, the reader is referred to the papers by Anderson (**1**), Southern California Edison Co., Ltd. (**10**), Duquesne Light Co. (**9**), Texas Power and Light Co. (**8**), Straub (**50**), Tray (**53**), Splittgerber (**48**), Goerke (**12**), and Hall (**14**). Sodium hydroxide, sodium carbonate, chloride, silicate, sulfate, and silica are reported in varying percentages. Splittgerber by diagram portrays the relative percentages present in the deposit from a turbine operating at 1800 psi. and 930 F. The deposit from the second stage of the turbine is characterized by more than 80 per cent of sodium chloride, 3 or 4 per cent water glass, and about 15 per cent of sodium hydroxide. At the ninth stage the sodium silicate is 10 per cent, sodium hydroxide is zero, sodium chloride 80 per cent and silica about 10 per cent. At the twenty-seventh stage of the turbine, silica alone is found. In general, the conclusion is definite that these soluble salts appear in the deposits from the earlier stages of the turbine but are largely or completely gone in later stages, being replaced by insoluble silica. Whether this condition in the latter stages is due to nonoccurrence of deposition, or rather to its removal because of enough moisture present to effect this, cannot be stated.

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[6] Private communication

According to Goerke (12), silica makes its appearance in the turbine at an upper temperature of approximately 535 F. Our observations show that it occurs in the form of quartz or amorphous silica as a function of the temperature and pressure. On an 18-stage machine, turbine throttle 730 F., between the eighth row at 572 F., and the twelfth row at 355 F., we noted that the heaviest silica deposit occurred on the tenth stage in the form of quartz. At the eleventh stage amorphous silica began to appear, and on the twelfth, thirteenth, and fourteenth stages the silica was entirely in the amorphous form. In another turbine, at the ninth stage with a temperature of 396 F., the deposit was quartz, and at the twelfth stage with a temperature of 233 F. was glassy, that is, amorphous.

Whether derived from the alkaline boiler water mechanically or by vaporization, certainly silica is present in the steam as sodium silicate and not SiO_2. How the transformation from sodium silicate to silica occurs is not yet recognized. Splittgerber (46) shows how the equilibrium of sodium silicate with carbon dioxide may represent a mechanism for the formation of silica in the latter stages of the turbine, crediting this theory to M. Zimmerman and W. Wesley in 1930. If that is the case, then removal of carbon dioxide from the steam ought to inhibit the breaking up of sodium silicate into a soluble component—sodium hydroxide or sodium carbonate—and insoluble silica. A turbine would then respond to washing merely with saturated steam in the case of any deposition on the blades, not requiring washing with sodium hydroxide which is being advocated to some extent at this time (12, 11), and which is necessitated by silica.

Our experience indicates that reduction of high carbon dioxide in the steam to very low values is of benefit, but that it is not a complete answer to the problem.

On the other hand, within the range of temperatures (also pressures) characteristic of the turbines, varying ratios of SiO_2 to Na_2O in the boiler water may result in the formation of $Na_2Si_2O_5$, Na_2SiO_3, and even SiO_2 (25) in the turbine. Work on the complete ternary system $H_2O—Na_2SiO_3—SiO_2$, which would give much information of value in this connection, has been started only by Morey (29), complete data not yet being available; the analogous system $H_2O—K_2SiO_3—SiO_2$ was completed by him (27) a number of years ago, and indicates clearly the transformations that may occur. Thus, there is the definite possibility of direct deposition of silica in the turbine in the transformations undergone by sodium silicate to establish equilibrium. Undoubtedly, similar equilibria governed the formation of two very interesting deposits that we have examined in the course of our work, one containing 52.3 per cent of silica, largely quartz, from the side wall tube of a boiler operating at 450 psi.; and the other from the dry pipe of a boiler operating at 125 psi., containing 63 per cent of silica which the polarizing microscope indicates to be largely in the form of $Na_2Si_2O_5$. The X-ray pattern[7] also shows lines which differentiate from quartz and metasilicate, and the significance of this is being studied at this time. So far as we know, this is the first instance in which this form of silica has been recognized in boiler-water work. The fact that the disilicate is less readily attacked by water (**25**) than Na_2SiO_3, and the limited moisture at the dry pipe to dissolve it, probably accounts for its discovery.

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[7] Examination by R. K. Scott, X-ray Specialist, Hall Laboratories, Inc.

HOW ARE SOLUBLE BOILER-WATER SALTS TRANSFERRED TO THE STEAM

Mechanical Entrainment.—As steam is generated from the boiler water, the spray engendered in the process may be entrained thereby and carried along therewith. This is the ordinary mechanical, conventional conception of carry-over. Whether considered to be foaming or priming, it is undoubtedly contributory to the impurities of the steam, regardless of what other processes also may be contributing thereto. To minimize it, various precautions are taken. Mechanically, these take the form of rather intricate baffling of the steam outlet.

Stability and regulation of height of water level in the steam offtake drum and its variation with load changes on the boiler are primary factors in minimizing carry-over, as demonstrated in a recent study by Rivers and Hill (**34**) at one of the large steel plants by means of steam purity tests. In relation to their effect on carry-over, total solids, sludge, saponifiable material, and alkalinity (**30, 11, 58**) should be classified together for their common property of causing disturbance of the water level. Quiescence in the steam drum is a factor in preventing carry-over, the study by Baker (**2**) on mechanical purification of steam illustrating what may be accomplished thereby. As stated above, the efforts to accomplish mechanical separation of boiler water from outgoing steam must suffer no letdown.

Vaporization of Boiler-Water Salts.—In 1933, on the basis of unpublished experiments of the Union of Large Boiler Owners, Lupberger (**21**) stated: "In experiments which extended up to 120 atmospheres, and to a salt content of the boiler water of 10,000 mg. per liter it developed that the salt content of the type NaCl, NaOH, Na_2SO_4 always amounted in the steam produced to some 2 to 3 mg. per liter independent of the boiler water level and of the loads. This amount of salt carries over, however, not with moisture, but volatilizes as salt itself corresponding to the partial pressure of the salt. The origin of this salt makes precaution in the boiler ineffective. Naturally, through foaming, the amount may be strongly increased. The two processes are, however, to be distinguished one from the other."

A little later Kleinhans (**17**) by calculation using the partial pressure and solution laws concluded that appreciable quantities of sodium chloride would enter the steam by vaporization in high-pressure boiler operation. No experimental work was done, and both Splittgerber (**44**) and Spillner (**43**) criticized the results very severely on the basis of overextrapolation of the known vapor pressure of sodium chloride in a higher temperature range, and thus of making the calculations on values not satisfactorily supported by experimental measurements. Splittgerber states: "Kleinhans considers boiler water a dilute solution and accordingly accepts the validity of the Raoult law therefor. Actually in his derivation he must look upon the boiler water as a concentrated solution of very much water in a small amount of melted salt."

Next, Koch (**18**) also by calculation on the basis of the solution and partial pressure laws derived amounts of sodium chloride and sodium hydroxide in steam by vaporization of a different order of greatness from those actually found, and concluded "that the salt content in steam from boiler water cannot be the result of vaporization."

Spillner (**43**) attacked the problem experimentally, with carefully constructed equipment, whereby superheated steam of known purity was caused to pass at a slow rate over solid salt so that, if the salt would vaporize into it, or in other words, dissolve therein, saturation might occur. In other experiments, saturated steam was passed over salt solutions. The steam was condensed, and measurement made on the solution for the concentration of the salt in question. The pressure range covered was from 1138 to 3229 psi. with limited experimentation at 4551 psi. The temperature of the superheated steam was 765 F. Salts used were sodium chloride, potassium chloride, sodium hydroxide, and sodium sulfate. The experiments with sodium sulfate were considered unsatisfactory because sulfate-containing decomposition products reacted to cause a high sulfate concentration in the distillate. No work was done on silicates. He found differing solubility in steam for the different salts NaCl, KCl, NaOH and Na_2SO_4, and that solubility increases sharply with increasing pressure. A conclusion of particular interest is that the concentration of salt in the steam is independent of the salt concentration in the boiler water. Spillner concludes that his results prove vaporization of salts to be the true cause of the salt-transport by high-pressure steam.

The data of Keevil (**16**), just recently available, throw some light on Spillner's work. At a temperature of 705 F., the solubility of sodium chloride according to Schroeder (**36**) and co-workers is 44.5 per cent and according to Keevil, the vapor pressure of this solution is 2100 psi. Thus, when Spillner passed superheated steam at 766 F. and the pressure range used in his experiments over solid sodium chloride, the sodium chloride was merely dissolved to form a solution which would be in equilibrium with the steam. Thus, the plan to saturate the steam by bringing superheated steam into contact with solid salt was sure to go amiss. Also, Keevil's observations on sodium sulfate when this salt was taken to the critical temperature and pressure of water without evidence of any decomposition or chemical reaction, are not in agreement with Spillner.

In our opinion, judgment on Spillner's work must be held in abeyance for the present.

While the paper by Cleve (**7**) is mainly a review, and accepts the 1136 psi. proposed by Spillner as the point at which vaporization into the steam begins to supplant mechanical carry-over, it cites a very interesting example of the effect of vaporization on the steam of a Benson boiler in which the steam contained 2 mg. per kg. of salt content which was the same as the salt content of the feedwater. This could not be explained by foaming in the separatory drum, but is explained on the assumption that the solubility in the steam was equal to the very low salt content of 2 mg. per kg. of the feedwater.

Finally, Splittgerber (**46**, **49**) has reported on the work done by Sauer in distilling solutions containing from 25 to 200 mg. per liter of silica at different alkalinities, both alone and in the presence of sodium chloride, sodium sulfate and sodium phosphate. Distillation was at pressures varying from 750 to 2250 psi. and was at the rate of about 1 liter in 3 hr., thus representing very gentle boiling conditions. Measurement showed a small amount of silica in the distillates, but on the basis of these figures Splittgerber concludes that, at least in most cases, increment of silica in the steam must occur not alone through vaporization of silica or silicate, but as well through mechanical carry-over.

In all of the work cited, consideration is given only to the conception of the boiler water as a whole, therefore as a quite dilute solution. All experimental work has been conceived on that basis, special care being exercised to insure gentle boiling conditions and to avoid the extreme rates of heat absorption and steam bubble formation which characterize the evaporative tubes of the modern boiler. Partridge (**32**), however, has taught that as the steam bubble forms, its procreating water reaches supersaturation in the process, leaving a ring of solid deposited boiler-water salt in evidence. Nor is this all. Markson (**22**) and his co-workers have shown that a hot section representing steam blanketing or film boiling may occur even in vertical evaporative tubes, with normal conditions in the tube both before and after the hot spot. Thus, each steam bubble as it is generated will approach equilibrium, not with the over-all boiler water but rather with a strong solution resulting from evaporating the boiler water to saturation, even to supersaturation, and in contact with a tube surface at a temperature at least equal to that corresponding to the saturated steam, and in many instances higher. True, concentrations established in the steam bubble by the environment of these conditions may be changed to some extent in its passage to the steam outlet. Since, however, all steam bubbles will bear the imprint of these conditions, we feel confident that the over-all steam at the steam outlet will represent a product of the temperature and saturation conditions existing at the point of formation of the individual bubbles rather than a product of the dilute over-all boiler water. We believe that any experimental work which will aptly duplicate boiler conditions must recognize these facts. Spillner's statement that the concentration of salt in the steam is independent of the salt concentration of the solution over wide limits, and Cleve's statement in regard to the Benson boilers, are in harmony with these considerations. Furthermore, the development frequently in the evaporative sections of the boiler of quartz-containing deposits by the transformation thereinto of sodium silicate in the boiler water is evidence of similitude of conditions in this region with those in the turbine where like transformations occur.

If we look at the problem of vaporization of boiler water salts into the steam in this way, and especially with the perspective of equilibria at critical temperatures and pressures as presented by Mr. Morey, the speculations in regard to transfer of boiler water salts into the steam by vaporization do not seem as farfetched as they do if we confine our thinking to the over-all dilute boiler water.

HOW MAY DEPOSITION ON THE TURBINE BE AVOIDED?

Pure steam generated from pure water is the obvious, but an impracticable answer. In some generating stations, an approach thereto is possible, and results have justified the procedure; in the great majority however, steam must be generated from waters containing varying amounts of dissolved salts.

In this majority of plants, therefore, the primary defense against turbine deposition must be to prevent as far as possible, any carry-over of boiler water in the steam. The means, as noted previously, are baffling of the steam outlet to provide complete separation of entrained boiler water from the steam; and, by control of water conditions, water level, etc., to hold to a minimum the amount of entrained water that must be separated from the steam by the baffles.

But now, if vaporization of boiler-water salts is of appreciable amount, it represents a source of impurity in the steam against which both water conditioning and baffling are futile. Also, frequently, the primary defense is not impregnable; impurities slip through, small in amount usually, but destructive of turbine efficiency.

Hence, an additional system of defense must be set up, namely, that of so adjusting the type of impurities in the steam that no condition of tackiness and hence of adherency to turbine blading will develop in the whole temperature range of the turbine. An alternative is to prevent formation of silica deposits, thus permitting simple washing of the turbine, or, in many instances, automatic washing thereof by moisture conditions arising through load changes or shutdowns.

Tackiness has been widely held to be the cause of adherence to the blades, and sodium hydroxide has been assigned the role of principal offender. Anderson (**1**), one of the pioneers of high-pressure steam generation, pointed to the fact that the silicate deposit began in his turbine at about 600 F. which is approximately the melting point of sodium hydroxide. In developing this idea, Straub (**50**) shows the extremely high concentration of sodium hydroxide in solution in equilibrium with superheated steam at the temperatures prevailing in the turbine (**19**). Tray (**53**), in agreement as to the part that sodium hydroxide plays, adds that when sodium silicate is also carried in the steam, the sodium oxide thereof adheres to the turbine blades exactly as does the sodium oxide from the boiler water alkalinity and in the same temperature ranges. Goerke (**12**) concludes that the deposition of soluble salt in the turbine is due mainly to the sodium hydroxide contained in the steam. Soderberg (**42**), commenting on Straub's theory, brings in a little different thought stating: "There is probably unanimity in the conclusion that scaling would not occur if the impurities remained as a dry dust. As the expansion proceeds to lower temperatures, however, it is entirely probable that other constituents than sodium hydroxide become sticky and facilitate scaling. In addition, there is some possibility that some of the scale is deposited by crystallization in a manner similar to that occurring when boiler tubes are scaling." Michel (**24**) notes that investigations in connection with the Benson boiler found that sodium hydroxide was the principal offender, especially in combination with silicate. Furthermore, in noting that salts, like alloys, form eutectic mixtures, he states: "Such salts carried over in solution from the boiler will lose partially or entirely their moisture content in the superheater. The remainder will pass through the superheater and into the turbine in a liquid state, either as droplets or as film on the superheater walls. The salt may deposit on the turbine blades as soon as the temperature becomes low enough for solidification to occur. Different deposits in different stages of the turbine can be explained by the different melting points of the salt or salt mixtures passing through the turbine." Buckland (**4**) brings in the application of negative-slope solubility curves stating: "The deposits would be expected to occur more thickly at points of rapid pressure drop, because the solubility of the salt decreases with pressure." Hall (**13**), discussing Buckland's paper, has emphasized further the effect of negative and positive slopes of solubility curves on the equilibria that will be established in the superheater, and then reversed as the steam passes through the turbine. Also, on the basis of data obtained in the research project on boiler feed water and embrittlement carried out by the A.S.M.E. and other technical societies in collaboration with the U. S. Bureau of Mines, Schroeder and co-workers (**36**, **35**) have pointed out that the negative solubility curves of sodium sulfate and carbonate become positive at temperatures above approximately 500 F. in the presence of dissolved sodium hydroxide whose solubility curve is positive in slope. As the changes of equilibrium are quite complex with decreasing temperature and pressure in the turbine, it is not surprising that sodium sulfate and sodium carbonate should appear along with sodium chloride and sodium hydroxide in the deposits therein. Sodium silicate as discussed earlier is a law unto itself.

All seem agreed that if tackiness of the impurities in the steam can be avoided in their passage through the turbine, adherence will not occur. How shall this be accomplished?

Straub (**50**) states: "Certain inorganic salts such as sulfate, chloride, and carbonate if present in sufficient amount will prevent this fouling. If sodium sulfate is to be used to prevent turbine fouling, it must be present in amount greater than four or five times the actual sodium hydroxide present."

Tray (**53**) states: "It is evident that silica cannot exceed 2 per cent of the total solids if insoluble deposits are to be prevented."

Werner (**56**, **57**, **45**, **47**) recommends appropriate treatment of feed and boiler water, to produce a salt mixture whose essential constituents do not melt in the temperature range of the superheater, and that do not congeal in the temperature range of steam expansion in the turbine, attaining this end by effective types and proportions of acids used to neutralize alkalinity in the water. Holding down the sodium hydroxide content alone without consideration being given to appropriate mixture of other salts is not enough—it will in general merely lower the amount of the deposition of salt in the turbine. Werner proposes a number of acids for his purpose, among them phosphoric, sulfuric, and sulfurous.

As to results from these different methods, difference of opinion exists. Buckland (**4**), for instance, recommends the Straub (**50**) procedure; Power Station Chemistry (**33**) reports a division of successes and failures. In application of the Tray (**53**) method, Splittgerber obtained no results. Wesley and Geisler (**58**) by severely lowering the caustic alkalinity in the boiler waters practically eliminated turbine deposition. Apparently the hydrothermal relationships of the customary boiler water salts in the superheater and turbine must be more thoroughly understood before the means of correcting the specific conditions of each plant can be chosen successfully.

Many gaps in existing data must be filled in before the complete story of turbine deposition can be written. It is evident that further exact work on the fact and quantity of vaporization of boiler-water salts must be done. Knowledge of the hydrothermal equilibria of boiler-water salts in superheater and turbine must be much expanded to permit any full solution of the problem involved.

MR. J. A. HOLMES[8] (*presented in written form*).—The study of solids in steam in an effort to find the causes and methods for elimination of deposits is an important item for power plants today. There is much to be learned about this subject, and the following observations are given to contribute data to those vitally interested in research on this subject. The observations are not presented as facts but are rather impressions gained from plant operating experience and results obtained from watching the action of small laboratory experimental boilers. The following is presented in as near outline form as possible to facilitate presentation and discussion:

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[8] Director of Service, National Aluminate Corp., Chicago, Ill.

I. HOW ARE SOLIDS CARRIED OVER?

1. *Mechanical carry-over* or entrainment of boiler water in steam is most prevalent at low pressures assuming boiler construction is such that there is not any jetting or syphoning action. Consequently, large steam space, baffles, or steam purifiers are of much help.

2. *Solution carry-over* (a term given by some for solids apparently dissolved or otherwise included intimately in the steam) becomes increasingly prevalent as pressure increases; consequently, mechanical separators at high pressures are of less advantage. *Washing* helps by concentration reduction, but does not necessarily entirely eliminate deposits.

3. *At what pressures do mechanical or solution carry-over occur?*

Apparently the main transition point is between 500 and 600 psi., although there is obviously a wide band of pressure over which both types may occur dependent also on construction or operation of the boiler and solids present in the boiler water. In experimental boilers, it is noted at about 500 psi. there is a tendency for the water in the boiler to quiet down with less ebullition of bubbles in the steam generating tubes, and, as the pressure increases, there is more surface boiling or direct change of the water to vapor at the surface. The steam or vapor at the lower pressure is transparent whereas the boiler water is nontransparent, while at higher pressures the steam is nontransparent and the water transparent.

4. Which affects solid content of steam —*pressure or temperatures?*

Pressure evidently plays an important factor for solids content of saturated steam leaving main part of boiler while temperature increase in the superheater may change character of solids and affect their degree of stickiness.

II. HOW AND IN WHAT FORM ARE THE SOLIDS DEPOSITED?

1. *Sulfate* content of deposits usually decreases as pressure increases.
2. *Silica* content of deposits usually increases as pressure increases.
3. *Silica* deposits vary from granular to smooth glossy coatings.
4. *Silica* deposits are occasionally found in highly hydrated form even though formed at high temperatures.
5. Occasional high *iron or copper* contents in deposits that apparently are not from turbines or equipment after the boiler.
6. Apparently more trouble is experienced with *superheaters* at low pressures and temperatures, the deposit containing large quantities of sodium chloride, sulfate, carbonate, and hydrate or washable salts.
7. As pressure and temperatures increase, tendency is for less deposits in superheaters and more in *turbines* with increase in nonwashable materials.

III. WHAT ARE EFFECTS OF DIFFERENT SOLIDS IN THE BOILER WATER?

1. It is difficult at present time to *correlate* solids in boiler water with deposits formed in superheaters or turbines.

2. There is some slight evidence of

selective carry-over of certain solids but more evidence of *selective deposition* depending on temperatures, velocities, and equipment.

3. *High chlorides* frequently have a depressing effect on carry-over—especially at lower pressures.

4. *Magnesium precipitates* apparently promote carry-over, especially that caused by foaming.

5. *High alkalinity* is more effective in causing carry-over than sulfates or chlorides, but is not always the determining factor.

6. *Silica* in steam increases as silica of boiler water increases. However, the silica content of the steam is not always the measure of amount of deposits that might form.

7. There is some evidence that the *ratio of alkaline solids to silica* in the boiler water, and, consequently, in the steam, may affect the stickiness of the silica. Since deposits occur at some plants with an extremely low silica content and not at other plants with high silica, possibly the presence of other materials such as dry solids or carbon dioxide may affect the alkalinity-silica ratio and reduce stickiness.

8. Finely divided *suspended solids* are more effective to promote carry-over at low pressures but evidently still have some effect at high pressures.

IV. PREVENTION OF DEPOSITS

(*a*) In general, *Straub's theories* for prevention of deposits work out very well.

(*b*) At *lower pressures* (say below 700 psi.) total solids concentration control of boiler water, use of baffles and purifiers and even antifoams help reduce solids in steam. To improve steam, concentrate mainly on preventing mechanical carry-over.

(*c*) At *higher pressures* washing helps as well as concentration control of boiler water solids. Until the deciding factor is known that causes stickiness or adherence, all solids in feedwater should be reduced to the lowest possible minimum.

V. CONCLUSIONS

1. Pressure or temperature or a combination of the two apparently affects the manner in which solids are carried by steam, that is, mechanical or in solution.

2. Concentration or amount of any one solid in boiler water or steam is not the deciding factor of whether deposits will form.

3. Apparently some other deciding factor, such as ratio of the various ordinary boiler water solids to each other, determines stickiness of solids in steam.

MR. N. L. MOCHEL.[9]—The building up of scale or salt deposits on the interior surfaces of steam turbines is of natural concern to the turbine builder. Any condition that lowers the efficiency or capacity of his product, or that may endanger its life, warrants his attention.

Just what the turbine builder may do about the matter is a question. He has and is studying designs to see if any help may be forthcoming from that angle. It does not appear that the selection of material of construction offers any great assistance.

Although we have been informed of cases of undue accumulation, and operators have discussed these problems with us, invariably the operator has found relief, or partial relief, or partial answer to the problem through cooperative study with the boiler manufacturer or the water-treating experts, and this would indicate that the ultimate solution or understanding of the problem lies in that quarter.

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[9] Manager, Metallurgical Engineering, Westinghouse Electric and Manufacturing Co., Philadelphia, Pa.

Nevertheless, the turbine builder recognizes in this problem a matter for cooperative research, largely between the operator and all those agencies that supply the steam for the turbine, but one in which he would be willing to join in any helpful way.

MR. F. R. OWENS[10] (*presented in written form*).—Spillner's (**43**) investigation of the behavior of certain pure inorganic salts in the pressure range of 1138 to 4551 psi. revealed that water does not suddenly lose its solubility for salts in changing from the liquid to fluid state at the critical point. He also showed that the solubility of certain inorganic salts in steam increases with increasing pressure.

General experience with high-pressure turbines, as Buckland (**4**) stated, finds depositions occurring to the greatest extent in regions of low pressure.

We recognize the probable impossibility of establishing equilibrium with the salts in boiler water and steam, in practice. However, we believe that our study of a number of high-pressure installations, in operation, does confirm some of Spillner's and Buckland's conclusions.

We believe the experience at one plant may contribute, at least in a small measure, to this general discussion. This plant has been in operation for three years and during this period it has been necessary to wash the turbines periodically.

The boiler is designed for 925 psi. and a temperature of 830 F. and delivers steam to a Curtiss compound turbine. We find, about 30 to 36 hr. after washing the turbine, a slight drop in capacity which requires an increase in boiler pressure. This gradually increases to the sixth to eighth day, at which time the capacity drop increases sharply and continues so until a reduction in capacity of $3\frac{1}{2}$ to $3\frac{3}{4}$ per cent is reached. This usually occurs within a period of six to seven weeks.

We find that during the first two days after washing the turbine the specific conductance of the saturated steam is of the order of 0.7 and 0.8 micromhos. The specific conductance then increases, as the pressure is increased, to maintain capacity, to a value which varies during the third to seventh week of 1.6 to 5.3 micromhos. A reduction in load does not affect the specific conductance unless it is sufficient to permit a reduction in pressure of 60 to 70 psi.

Our test runs reveal that within the limits of 300 to 550 ppm. total dissolved solids there is no noticeable difference in specific conductance of the saturated steam at full load. The silica as SiO_2, with the above total dissolved solid range, will vary from 9 to 13 ppm. Also, there is no difference in the time elapsed until washing is necessary upon a basis of a capacity drop of $3\frac{1}{2}$ to $3\frac{3}{4}$ per cent. Concentrations above 550 ppm. total dissolved solids, and especially 700 ppm. and higher, result in an increase in specific conductance of the saturated steam.

We have maintained higher concentrations of Na_2SO_4 to conform to Straub's (**50**) ratio without increasing the time between washings. An identical experience is cited in our discussion (**31**) of Buckland's paper on "Turbine Nozzle and Bucket Deposits."

We have found it possible to increase, slightly, the time between washings by maintaining lower total alkalinities, the most favorable range being 110 to 140 ppm. in terms of $CaCO_3$. The pH would approximate 10.5 to 10.7 and the PO_4 and total dissolved solids 40 to 45 and 280 to 320, respectively.

We encountered difficulty at the subject plant due to a dry area in one row of tubes. This resulted in a large amount of magnetic oxide, both in suspension and adhering to the evaporating surfaces. Usually, after returning the boiler to service, and irrespective of whether it had or had not been drained, this suspension was quite heavy. We also find that as the suspended Fe_3O_4 decreases the silica decreases. When the suspension is a maximum the silica in the boiler water approximated 37 to 40 ppm. and gradually decreased to 9 to 13 ppm. as the suspended iron oxide became negligible.

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[10] Secretary, Cyrus William Rice and Co., Inc., Crafton, Pa.

Our observations show that in certain high-pressure installations using sea water for cooling no deposits are encountered in the turbines. At another plant with the same load, pressure, and temperature, but using fresh water for cooling, deposits do occur. We are now investigating the possibilities of the addition of magnesium and iron salts but the work has not progressed sufficiently to discuss the results at this time.

We can summarize our observations as follows:

1. Maintaninig Straub's ratio of Na_2SO_4 to NaOH does not, in all cases, eliminate turbine deposits at high pressures.

2. The solubility of certain inorganic salts increases in saturated steam as the pressure increases.

3. There is a definite influence exerted by certain salts on the solubility of silica in steam in its generation at high pressures.

4. This influence is not always the same in different installations of identical pressure and load and may be due to both a difference in chemical and physical environment.

5. In the pressure range cited, there is mechanical entrainment as evidenced by the presence of Fe_3O_4 on the turbine buckets. We suggest, therefore, the probable influence of dry areas in high-pressure operation with respect to turbine nozzle and bucket deposits.

MR. J. B. ROMER[11] (*presented in written form*).—Silica exists in natural waters in varying forms and amounts. In certain instances, the silica is present in suspended matter, but for the purpose of this discussion we are disregarding this form and confining our remarks to that silica which may be present either as a true solution of silica, or a silicate, or as a colloid.

Silica has always been a constituent in boiler scales. However, its rise to prominence occurred only after various improvements in boiler station equipment and practice and in water treatment reduced the other troublesome materials to such a level that silica became a predominant percentage of the total troublesome material.

During recent years, we have studied silica and silicate scales in boilers and turbines from a number of angles and have considered very carefully the theories advanced by various geologists and geophysicists regarding the formation of certain silicates as found in the earth's crust.

The principal thoughts gleaned from the literature and from conferences with various geologists indicate that under pressure hot aqueous solutions containing silica and alumina are capable of forming complex silicates at relatively low concentrations of dissolved solids; further, that little metamorphism occurs under the action of dry heat. Instead, recrystallization and silicate formation are more dependent upon water solutions. It has been pointed out that compounds like aluminum silicate and hydroxides of iron are characteristically absorbent of many salts prior to transformation into crystalline compounds. In connection with the formation of zeolites, it has been stated that the colloidal silica and clay in the mud react with the ferric iron set free by oxidation and the precipitated gel of iron silicates absorbs potassium or sodium salts from the water. It is interesting to read the literature on the synthetic production of montmorillonite wherein it is shown that the ratio of the various ingredients added markedly affects the character of the complex silicates formed. By adjusting the ratio of sodium and aluminum oxides and silica and heating to 572 F. with sufficient water to give a pressure of 1280 psi. in a platinum crucible in a steel bomb, it was possible to produce analcite.

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[11] Chief Chemist, Babcock & Wilcox Co., Barberton, Ohio.

Straub has published an article dealing with the synthetic production of analcite (**49a**) showing that he was able to produce a material having all the properties of the natural analcite.

It becomes quite evident after studying the literature that analcite formation is brought about by maintaining certain ratios between the alkali, alumina, and silica. That these conditions frequently exist in modern high-pressure boilers is evidenced by the fact that we frequently encounter analcite, and its elimination has been accomplished by changing the constituents present in the feed and boiler waters.

In view of the belief that various members of the zeolite family have been formed as a result of hydrothermal conditions, it becomes quite possible to consider that the form in which silica is deposited on a turbine blade is controlled by the hydrothermal conditions. This leads us to the question: how does the silica reach the turbine?

Spillner in his studies which led to the paper on "High Pressure Steam as a Solvent" (**43**) has gone to great lengths in an effort to obtain fundamental data on this subject, and I consider that a great deal of credit is due Spillner for his painstaking efforts along this line.

Spillner's paper shows that he has based his work on Dalton's law and on the known fact that all substances exert a vapor pressure, the magnitude depending upon temperature and the specific material. In most cases this vapor pressure below the melting point is of a very low order of magnitude and generally it does not become of a high order of magnitude until the substance has been heated to some temperature between its melting and boiling points. Spillner has made considerable point of the fact that Hannay and Hogarth (**15**) observed discoloration by bromine and iodine and certain halides of the vapor phase of alcohol when under pressure. These are elements and compounds all notable for their low melting points and high vapor pressures.

While we are inclined to agree with the theoretical considerations which led Spillner to his final conclusions, we cannot agree that the practical operating experience with modern boilers in any way supports the final conclusions reached by him.

We believe that one of the major causes for the difference lies in the fact that he used superheated steam, whereas in boiler operation any carry-over of solid particles to the steam must occur at saturation temperatures as the steam traverses some distance after leaving the drum before entering the superheater. In general our experience has indicated that the carry-over, in parts per million, falls in a narrow band when plotted against pressure, whereas Spillner shows it to be in the form of a curve rising very sharply with rise in pressure. This fundamental difference between Spillner's work and our experience is well illustrated by the following.

During the past year the Babcock & Wilcox Co. has completed a survey of 16 utility stations having 41 boilers equipped with cyclone separators and steam scrubbers and having a total capacity of 17,600,000 lb. of steam per hr. at operating pressures from 700 to 2400 psi. which have been in operation for various lengths of time up to 4 yr. This survey was conducted to obtain their operating experience with regard to turbine blade deposit and its effect on capacity and efficiency of the turbine units.

In all of these plants, the range of solids carry-over in the steam was between 0.3 and 0.7 ppm. as measured by the accepted conductivity method. For a given boiler, the solids carry-over in the steam would remain constant within a 0.1 ppm. regardless of the steam output from the boiler or the boiler water concentration within the limits covered during the tests which covered a wider range in every case than that experienced during regular operation.

Eleven of these stations have not experienced any noticeable loss of capacity or efficiency and do not wash the turbines. This indicates that the quantity and quality of the solids carry-over together with their load schedules and designs are such that the deposits do not reach a stage where operating results are affected.

Of the five remaining plants, one operated for a period of 24 months before noticing any loss of capacity on the turbine. The turbine was washed in service and the capacity restored. This was a 1400-psi. pressure unit operating with a boiler water concentration of 2000 ppm. Shortly thereafter the impulse stages were installed in the turbine and at the same time the boiler water concentration was reduced to 1100 to 1300 ppm. Since that time subsequent operation has shown no loss in capacity or efficiency and washing of the turbine has not been resorted to. In this particular case no measurable difference in the solids carry-over in the steam was noted when the boiler water concentration was reduced from 2000 ppm. down to the lower figure.

Another of these five stations which operated with 100 per cent make-up of water high in silica loses approximately 3 per cent in capacity over a three-month period. This is restored by washing the turbine in service on a Sunday every three months. No insoluble deposit which affects capacity or efficiency is experienced.

Another of these five plants which has a solids carry-over of 0.4 ppm. experiences a 7 per cent increase in the first stage pressure of the high-pressure unit over a 60-day period. The stage pressure is brought back to normal at the end of this operating period by dropping the steam temperature from 900 F. down to 750 F. for 2 to 3 hr. No insoluble deposit which affects operating results has been experienced.

In the remaining two plants a gradual loss of efficiency was experienced over an operating period of 1 yr. and examination of the turbines showed insoluble silica deposits.

MR. F. G. STRAUB.[12]—About 10 yr. ago we were asked to make a study of the problem of turbine blade deposits. This was due primarily to the occurrence of deposits in the turbines in the plants of our sponsoring companies.

At that time, with the cooperation of the Prime Movers Committee of the Edison Electric Institute, we obtained a large amount of data relative to the type and composition of many turbine blade deposits. Luckily for us we were able to divide the type of blade deposits into two classes, water soluble and water insoluble, the insoluble type being mainly high-silica material. Rather fortunately for us, the type of deposits which was occurring at that time in the turbines of the plants of our sponsoring company was of the water-soluble type.

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[12] Professor of Chemical Engineering, University of Illinois, Urbana, Ill.

We built a small boiler in which we generated steam, contaminated the steam with various chemicals, superheated the steam and passed it over a stationary turbine blade. We found salts which would cause appreciable deposits, and salts and a combination of salts which would not. At that time, we found that a high sulfate to alkalinity ratio in the boiler water appeared to prevent deposits in our laboratory work. We applied this type of treatment to several plants in the Chicago area and obtained favorable results.

We were able to divide the type of deposits into water soluble and insoluble, and at that time we said that we were interested only in the water-soluble type and sidestepped what is now the major problem of the blade deposits. About 2 yr. ago, our sponsoring companies again raised the question of preventing turbine blade deposits. I do not think it was primarily due to the fact that they were having trouble, but that they were seeing other plants in trouble, and since they were contemplating going to higher pressures, they wanted to prevent any occurrence of trouble in the future.

We started the new investigation about 2 yr. ago with the idea of extending our previous work which had terminated at 600 psi. pressure and 700 F. to the critical pressure and 1000 F. We built a small laboratory boiler similar to what we had done before. About the time we were starting this work we happened to run across the publication by Spillner (**43**). In this article he stated that there was a definite solubility of various salts in steam at high pressures.

While I consider Spillner's research a very good piece of work, I believe it has delayed our work more than it has aided us, since we have not been able to reproduce Spillner's results. At 2000 to 2400 psi. pressure we have not been able to obtain a salt concentration in saturated steam much greater than 1 ppm., and most of the time it is in the order of tenths of a part per million.

We hesitate to pass over a piece of work of the nature that has been done by Spillner and say that it is wrong without discovering the fundamental cause of the difference between his and our results. We have not been able to answer this question to date. I believe, however, that in the past few weeks we seem to have an indication that the main reason for the difference appears to be in the fact that undoubtedly Spillner, though he reported results of both superheated and saturated steam, had superheated steam in both instances. If this was so, he would have superheated steam in contact with solid salt which had formed on his purifying plates and would obtain the equilibrium condition existing between the solid salt and superheated steam. With saturated steam in contact with a dilute solution of salt the solubility of the salt in the steam will be much lower. We are continuing the research to obtain further solubility data. It is possible that his saturated steam was superheated.

I should like to take this opportunity to bring one fact to your attention. I have been trying to sell this idea for the past 10 yr. to the power plant operators, and apparently I am not a good salesman. When we did our early work on turbine blade deposits, we developed a small test unit that we installed in the power plant. It involved nothing but an orifice and a couple of stationary blades, and we took a small sample of the steam right ahead of the turbine and passed it over these turbine blades under definite pressure and temperature control. At frequent intervals we removed these blades and examined them for deposits.

In several plants we were able to make a study of the blade deposits and show a definite deposit in periods of 50 or 75 hr. of operation. I believe that it would pay for the operators to devise some kind of small test unit of this type which could take steam from various stages of their turbines (particularly from the stages in which they are having trouble) and pass it through an orifice, impinge on a blade, and in a short period of operation see whether they are having trouble or not. If they are, they can then make a study of the conditions and see whether they can eliminate the trouble.

MESSRS. S. E. TRAY[13] AND C. E. IMHOFF[13] (*presented in written form*).—The occurrence of chemical deposits on turbine blading is one of the major problems encountered in the generation of power from steam. More particularly, this problem manifests itself in superimposed turbines and high back-pressure turbines both in central stations and industrial plants. The effect on turbine efficiency and over-all economy is more pronounced with these units than with straight condensing turbines. Operation on the distilled water cycle of a central station, as contrasted with the high make-up requirements of industrial plants supplying process steam, does not appear to reduce the tendency for these deposits to accumulate.

The boiler manufacturers have approached the problem of producing clean steam from the standpoint of reducing the amount of solids carried over in the steam. Considerable progress has been made in this direction through the medium of steam washers, baffles, screens and other mechanical devices. Steam with a solids content of the order of 1 ppm. or less is readily obtained in commercial operation. That this has not eliminated turbine blade deposits is an indication that the nature of the solids in the steam, rather than the amount present, is the important consideration. A study of various deposits actually encountered in superheaters and turbines leads to the conclusion that mechanical carry-over, except in certain instances, cannot be the sole explanation.

Occurrence of Deposits:

Chemical deposits occur in superheaters and in turbines throughout the entire range of superheated steam temperature. They are not found below the dew point in the turbine. They may be classified as soluble or insoluble deposits depending on whether they may be washed off the turbine blades with saturated steam. The chemical composition of the deposits varies according to the temperature zone in which they are laid down. The prevalence of one compound over another in blade deposits has been considered by some to indicate preferential carry-over from the boiler. Actually this may result from the concentration of certain boiler water salts in foam bubbles due to the effect of surface tension.

In the study of turbine blade deposits, the authors have obtained considerable data from X-ray diffraction studies of a large number of samples. It is noteworthy that in practically all cases these deposits are crystalline and have a definite crystal structure. From this evidence, which includes a study of crystal size, there can be no question but that the deposits were formed by precipitating from solution at the point where they were found on the turbine blades. The inference follows naturally that the salts were originally dissolved in the steam and came out of solution when the steam became supersaturated with respect to a particular salt. The accompanying Table I shows the temperature range in which various salts have deposited in superheaters and turbines. It is suggested that these follow the order of solubility of the salts in superheated steam.

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[13] Manager, and Chemical Engineer, respectively, Boiler Feedwater Division, Allis-Chalmers Manufacturing Co., Milwaukee, Wis.

Crystal Formation:

The presence of crystals in turbine blade deposits leads to a consideration of the mechanism of crystal growth. Thermodynamically, there are a number of phenomena which play a part in the birth of a crystal from a solution phase. Every change in phase is accompanied by changes in thermal energy. Crystals forming from any solution must pass through energy phases corresponding to heat of dilution, heat of solution, surface tension and interfacial tension between liquid and solid. Factors governing crystal formation and growth are independent of whether growth proceeds from the vapor, molten or solution state. The two essential conditions necessary for crystal formation are: (1) the existence of a supersaturated solution, and (2) the presence of nuclei or seed crystals. Supersaturation refers to the condition existing when a substance has exceeded its normal solubility concentration. Such a system is under great internal stress and strives toward a lower state of energy. The avenue for relieving this energy is through crystallization, but nuclei are needed to initiate the crystallization. Nuclei or seed crystals need not be identical with the substance in the supersaturated solution but may be closely isomorphous, isostructural or of the same nature and still have the power to induce crystallization. In the case of turbine deposits, microscopic irregularities on the surface of the blades may serve as nuclei for crystallization or as points for initial crystallization.

TABLE I.—TEMPERATURE RANGE IN WHICH VARIOUS SALTS HAVE DEPOSITED IN SUPERHEATERS AND TURBINES

Sodium sulfate (Na_2SO_4)	900 to 700 F.
Sodium chloride (NaCl)	700 to 600 F.
Sodium hydroxide (NaOH)	600 to 550 F.
Sodium carbonate (Na_2CO_3)	600 to 550 F.
Sodium silicate ($Na_2O \cdot XSiO_2$)	600 to 550 F.
Silicate (quartz) (SiO_2)	550 to 450 F.

Prevention of Deposits:

In general, the theory of the solubility of boiler water salts in steam can very well account for the crystallization of turbine blade deposits as they have been observed to occur. Such deposits are most likely to be prevented by maintaining the salt content of the saturated steam from the boiler below the solubility of the various salts at turbine temperatures. Prevention by influencing crystal habit or using surface action is not promising. To be effective, surface active agents should be adsorbed strongly by all the salts present in turbine deposits. This seems unlikely in view of the high degree of specificity of most adsorbents and adsorbates.

A method of preventing turbine deposits by manipulating the ratios of salts in the boiler water has little support from the rules of crystallization. Straub (**50**) has suggested and Buckland (**4**) has reported specifically on the successful elimination of turbine blade deposits in a central station by maintaining certain ratios of Na_2SO_4 to NaOH. In view of the failure of this method to produce results in several other instances, it is suggested that the successful results were accomplished by influencing crystal growth in some manner not readily apparent.

Supersaturation cannot be influenced

unless certain of the salts are mutually soluble and produce an eutectic whose melting point is either higher or lower than the temperature range of the turbine. No such required solubilities are known to exist. Silica and sodium hydroxide are somewhat mutually soluble, but pure sodium hydroxide and pure silica are found to crystallize out in overlapping temperature zones within the turbine. Various ratios of silica and sodium hydroxide, therefore, do not shift the primary deposition zones. Sodium sulfate is not capable of changing the crystal habits of any salts normally found on turbine blades, and there is the further objection that attempts to increase sulfate may result in superheater deposits.

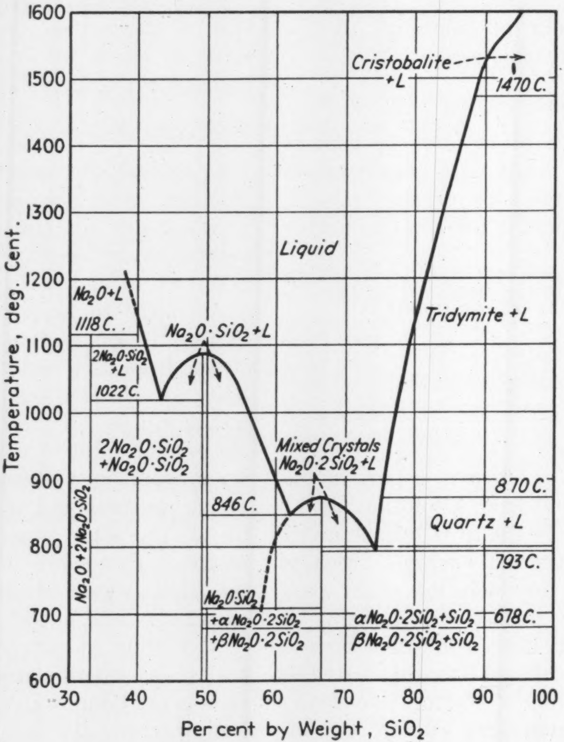

FIG. 1.—Kracek's Equilibrium Diagram of the System Na_2O-SiO_2.

Insoluble Blade Deposits:

A large percentage of the insoluble turbine blade deposits are composed of alpha quartz. Those composed of sodium silicate are water soluble and susceptible to removal by washing with saturated steam. Since blade deposits crystallize out of a solution phase, the various components should be present according to equilibrium conditions. If this is true, then the composition of the various phases present can be predicted by equilibria diagrams. A study of the system $Na_2O - SiO_2$ as described by Kracek (20) leads to some interesting conclusions. The components of the system as indicated in the accompanying Fig. 1 are Na_2O, Na_4SiO_4, Na_2SiO_3,

$Na_2Si_2O_5$ and SiO_2. The ratios of Na_2O to SiO_2 determine what components are in equilibrium. The accompanying Table II shows the stable components which exist.

This table indicates that quartz cannot be formed if the SiO_2 — Na_2O ratio is less than 0.67. In terms of sodium hydroxide and silica, the NaOH — SiO_2 ratio is 1.92. The inference follows that blade deposits in which the sodium hydroxide content is at least twice the silica are water soluble. Translating this to boiler water analyses, quartz will be formed on turbine blades if carry-over takes place from a boiler water in which the sodium hydroxide content is less than twice the soluble silica content. The converse is not true, because there is some evidence to support the thought that silica and sodium hydroxide do not carry-over in the steam in the same ratio as they exist in boiler water. A study of the NaOH — SiO_2 ratios of boiler water and steam in plants experiencing both soluble and insoluble turbine blade deposits will be necessary to determine the practical utility of this theoretical ratio. Some modification of Kracek's diagram is probably required in order to be applied to the composition of turbine blade deposits. This is due first of all to the fact that equilibrium conditions may not have been attained at the blade surface, and, secondly, the high partial pressure of steam may cause hydrated salts to be the stable phases, instead of the anhydrous salts indicated in the diagram.

TABLE II.—STABLE COMPONENTS.

SiO_2 in System Na_2O–SiO_2, per cent by weight	Phases Present
0 to 33	Na_2O and Na_4SiO_4
33 to 49	Na_4SiO_4 and Na_2SiO_3
49 to 66	Na_2SiO_3 and $Na_2Si_2O_5$
66 to 100	$Na_2Si_2O_5$ and Quartz

Surface of Tension:

Surface tension of boiler water is seldom mentioned in connection with turbine blade deposits in spite of their obvious relationship. Detergents such as sodium hydroxide, sodium phosphate and sodium silicate have an appreciable effect in reducing surface tension. A liquid in which the surface tension is reduced is prone to foam, especially with foam stabilizers present.

Gibb's adsorption law states that if a substance lowers the surface tension, that substance becomes more concentrated at the interface. In a boiler, a lowering of surface tension promotes a certain amount of foaming. The surface tension reducing agents are more concentrated in the foam bubbles than in the main body of boiler water. The composition of the carry-over, therefore, will be more like the composition of the foam bubbles than of the boiler water. In this manner, the apparent preferential carry-over of certain boiler water salts can be explained. Further study is required on methods of determining the concentration of individual boiler water salts in steam. Total dissolved solids can be determined quite readily by conductivity methods, but micro methods will have to be applied to the determination of individual salts. Some progress has already been made in this direction by the development of a method for the determination of silica in steam down to a concentration of 0.02 ppm.

Type of Deposits:

A number of crystalline constituents have been identified in turbine blade deposits. In general they are usually composed of the soluble salts in boiler water. Exceptions, however, are found in the case of calcium salts such as the carbonate and phosphate. These represent the precipitated sludge in boiler water which was apparently carried over mechanically rather than being dissolved in the steam. The obvious explanation is that these types of deposits result from an actual priming condition in which slugs of boiler water are lifted over to the turbine.

Silica is found both as soluble sodium silicate and as insoluble quartz. In the case of soluble silica, it has thus far not been possible to identify by X-ray methods any of the silicates which normally would be expected to form. Nevertheless, since many silica deposits are actually water soluble, it must be assumed that they exist as one of the several salts of silicic acid. Quartz has been found as a soft powdery deposit as well as a hard glass-like substance. The X-ray patterns of both forms are identical and they are equally insoluble in water. Their physical characteristics, however, are much different but thus far there has been no explanation to account for this difference. One instance of amorphous silica deposits has been noted in a turbine operating at 1400 psi. Further study is being made of the conditions accompanying the formation of this type of silica deposit on turbine blading.

Removal of Turbine Blade Deposits:

Any discussion of turbine blade deposits involves the question of removing these deposits. Normally, soluble deposits have been washed away with saturated steam or by soaking with water, while insoluble deposits are removed by sand blasting or scraping with the cover lifted and the turbine out of service. As further proof of the solubility of boiler-water salts in superheated steam, the authors have developed an interesting application of the theory for the removal of turbine blade deposits without interfering with normal turbine operation.

As has been previously indicated, the various boiler-water salts deposit in the turbine in the order of their solubility in steam. Accordingly, sodium sulfate which is found in the highest temperature zone, is the least soluble in steam. In the same manner, silica in the form of alpha quartz is found in the lowest temperature zone above the dew point, and hence is the most soluble in steam. With this conception in mind, it is at once apparent that if the temperature at any particular stage in the turbine can be raised above the solution point of the salt deposited at that stage, then the salt will redissolve in the higher temperature steam. With this method, sodium sulfate, because of its low solubility in steam, should be difficult to remove, while quartz should be readily dissolved due to its higher solubility. Fortunately sodium sulfate is seldom found in turbines to any extent, while silica in the form of quartz is quite prevalent and is the most difficult to remove by ordinary methods.

Experience with the practical application of this method of removing turbine deposits has been very satisfactory during the past year. One case of interest is an 800 psi., 800 F. topping turbine installed in a plant with other similar units, all operating under the same steam conditions. The turbine is a 16-stage machine exhausting at 235 psi. and 550 F. The turbine is equipped with a by-pass valve which permits the introduction of steam at throttle temperature to the third inlet valve following the sixth stage. When deposits were found to be limiting the capacity of the turbine, it was determined that they were present on the last four stages. They were also found to be of a soluble nature, low in silica, and composed of a mixture of boiler water salts. The accompanying Fig. 2 shows the temperature curve of the turbine for normal

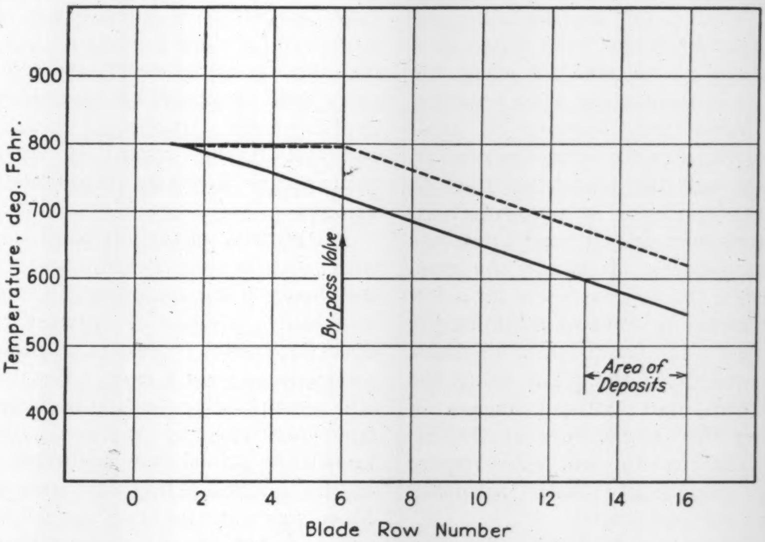

FIG. 2.—Effect of By-Pass Valve on Turbine Temperatures, 800 psi., 800 F., 240 psi. Back Pressure.

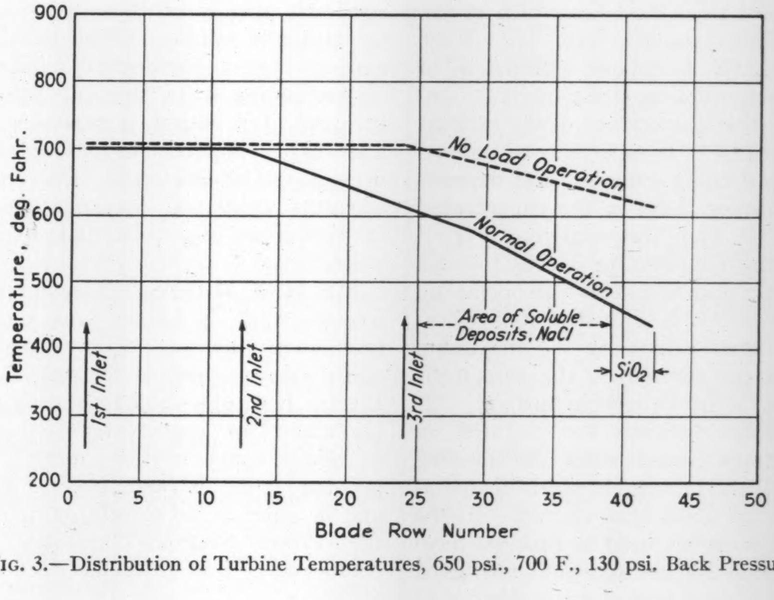

FIG. 3.—Distribution of Turbine Temperatures, 650 psi., 700 F., 130 psi. Back Pressure.

operation and the effect of opening the by-pass valve on the temperature. All of the last four stages were raised to a temperature above 600 F. since the exhaust temperature was approximately 615 F. With this increase in temperature, the deposits were dissolved in the steam and the blades returned to their clean condition as was evidenced by an increase in capacity and a decrease in stage pressures. It is now the practice to open the by-pass valve for a few minutes each day, thus maintaining the turbine in a clean condition at all times. Other turbines in the plant using the same steam, but without means of increasing the temperature at the exhaust, continue to foul and require washing periodically with saturated steam at reduced loads.

In the case of insoluble silica deposits, the same effect has been obtained. A 650 psi., 700 F. turbine has 42 stages and exhausts at 130 psi., 435 F. Soluble deposits, principally sodium chloride, are formed from the twenty-fifth to the thirty-ninth stage. The last three stages have a deposit composed of 85 percent silica as alpha quartz. Increasing the temperature of the exhaust to about 610 F. by operating at no load for several hours removes these deposits by dissolving them in the superheated steam. The accompanying Fig. 3 shows the temperature curves for this unit under normal and no-load operation.

This method is limited in its application to some extent by the maximum temperature allowed on the equipment following a back-pressure turbine. If low-pressure turbines are operated on the exhaust steam from the topping unit, a slight increase in steam temperature is of little consequence. If the exhaust steam is used in process, however, the equipment may not be designed for the higher temperatures that may result. In such cases, particularly when operating at no-load, the turbine may be permitted to exhaust to atmosphere for short periods necessary to maintain clean blade surfaces. No difficulty will generally be experienced in applying this process to condensing turbines, if due regard is given for temperature increases throughout the machine.

The removal of turbine blade deposits with superheated steam substantiates the theory of the solubility of these salts in steam. While of interest to the operator, such a process is, at best, only a remedy and not a cure. The problem still remains of preventing such deposits from occurring by application of the knowledge gained from a further study of the solvent action of water vapor. More fundamental data on solubilities are required in the temperature and pressure ranges normally encountered in power plant operation. A study of the solid phases of the various salts present in steam would be helpful, as well as equilibria studies of those salts which are mutually soluble. With this information at hand, together with a thorough understanding of the physical chemistry involved, it is entirely conceivable that a means of completely preventing turbine deposits can be developed. The problem warrants continued investigation and the best efforts of chemists and physicists alike.

MR. H. C. MILLER.[14]—I should like to inquire what is being done on this problem in the way of any organized effort. As a member of the Edison Electric Institute, which represents possibly about the largest organized group of power users in the country, this problem is of extreme interest to us, and in order to do something about it, the Power Station Chemistry Subcommittee of that group has been working for the past two or three years on the development of a very comprehensive questionnaire. This questionnaire is designed to get some of the case history on this very important subject, and, to give you an idea of its scope, comprises somewhere between 12 and 14 double-sized typewritten pages. It is being sent to 50 member companies of the Edison Institute.

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[14] Chemical Division Chief, Testing Laboratory, Public Service Electric and Gas Co., Maplewood, N. J.

The type of information asked for not only deals with chemical aspects of water in both the fluid and gaseous state but also goes quite extensively into some of the mechanical aspects of the problem. Because of its scope and the huge amount of data which we know will be accumulated, no conclusions will probably be available for quite a long time. Nor do we have any hope that we shall arrive at any immediate solution or any formula that will be universally helpful.

We do hope that we will have found out some of the common conditions that contribute to carry-over and possibly narrow down to a certain extent the range of temperature and pressure which includes the more extreme cases.

MR. M. D. BAKER.[15]—Mr. Morey's paper incites various thoughts concerning steam quality, especially regarding the possibility of eliminating that portion of the carry-over that causes silica deposition on the turbine blades. Until recently, carry-over in steam was considered entirely as mechanical entrainment, but with the advent of several papers, such as Spillner's (**43**), the thought of inorganic chemicals being carried over in the form of vapor has been presented. In the study to determine in what form the boiler water chemicals are conveyed out of the boiler, this discussion is offered. The data presented are based on steam quality tests, turbine washings, and analyses of turbine blade deposits.

The boilers at Springdale Station and the method used for determining steam quality have been published (**2**). With boiler drum pressures of 1350 psi. and boiler water concentration of 2000 ppm., steam is delivered during normal operation having 0.25 ppm. or less concentration. As 1,400,000 lb. of steam pass through the high-pressure turbine each hour, this concentration means that 7.8 lb. of material are carried to the turbine each day. The retention of a small portion of this amount would soon affect the efficiency of the unit.

TABLE III.—ANALYSIS OF DEPOSITS REMOVED FROM TURBINE BLADES.

	Sample No. 1, collected 1st min.		Sample No. 2, collected 2nd. min.		Sample No. 3, collected 5th and 10th min.	
	ppm.	Percentage of Total	ppm.	Percentage of Total	ppm.	Percentage of Total
Total solids	9930	...	17 300	...	2534	...
Suspended solids	100	...	1 830	...	326	...
Dissolved solids	9830	...	15 470	...	2208	...
Analysis of dissolved solids:						
Silica	2998	30.50	4 885	31.58	256	11.59
Iron and aluminum oxides, $Fe_2O_3-Al_2O_3$	51	0.52	49	0.32	149	6.74
Calcium, Ca	11	...	3	...	5	...
Magnesium, Mg	9	...	6	...	trace	...
Phosphate, PO_4	228	2.32	345	2.23	99	4.48
Sulfate, SO_4	1520	15.46	2 658	17.18	912	41.30
Chloride, Cl	380	3.87	100	0.65	8	0.36
Hydroxide, OH	214	...	267	...	15	...

Sufficient material was retained on the spindle of the high-pressure turbine to cause a 10 per cent decrease in capacity, so the turbine was washed with saturated steam in August, 1940. The washing was made with 350-psi. steam at saturation temperature. While reducing the superheat, the condensate of the steam passing through the turbine contained less than 1.0 ppm. solids. When saturation temperature was reached, the concentration of the condensate increased to 9930 ppm. the first minute, 17,300 ppm. the second minute and reduced to 2530 ppm. during the fifth to tenth minute of washing. Following this, the concentration decreased rapidly so that at the end of 30 min. from the start of the washing, the condensate contained less than 10 ppm.

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[15] Chief Chemist, West Penn Power Co., Springdale, Pa.

The accompanying Table III gives an analysis of the soluble material removed from the spingle (sodium was not determined). Also, it illustrates how the salts varied in concentration at different periods of the washing. The percentage of silica was about constant during the first two minutes, but diminished towards the end. The iron and aluminum oxides were much higher in the last sample, the phosphates were highest in the last sample, the sulfates increased in each sample and were much higher in the last sample and the chlorides decreased appreciably after the first sample.

TABLE IV.—ANALYSIS OF UNDISSOLVED MATERIAL REMOVED DURING WASHING.

Silica SiO_2, per cent	31.5
Iron and Aluminum Oxides, Fe_2O_3—Al_2O_3, per cent	40.4
Calcium Oxide—CaO, per cent	3.9
Magnesium Oxide—MgO, per cent	0.1
Sulfur Trioxide—SO_3, per cent	2.3
Phosphate—P_2O_5, per cent	3.9
Loss on ignition, per cent	18.0

The accompanying Table IV gives an analysis of the undissolved material which is mostly silica and iron and aluminum oxides.

After washing, the spindle was not inspected, but the turbine was restored to normal capacity. An inspection of the spindle some months later showed it to be clean. During the time interval between the washing and the inspection, full load could be carried on the unit.

Washing a low-pressure turbine, 350 psi. did not give the same beneficial results. After having lost about 17.5 per cent of its capacity, the turbine was washed with saturated steam and 7.5 per cent of the capacity was restored. The other 10 per cent was not obtained until the turbine was dismantled and the spindle sand-blasted.

The highest concentration attained by the condensate during this washing was 514 ppm., of which 100 ppm. was undissolved material. Partial analyses of the dissolved and undissolved material removed from the turbine blades are contained in the accompanying Table V.

After washing, the turbine was dismantled and an inspection of the spindle showed the two impulse rows and the first four rows of reaction blades to be clean. Starting at the fifth and up to the ninth row, the reaction blades were coated with a brittle deposit. The eighth row contained the heaviest deposit. A microscopic analysis of this material showed it to be mostly quartz crystals. The chemical analysis was 96.5 per cent SiO_2. The tenth row was almost clean. The eleventh to the sixteenth rows were coated with a powder-like deposit which was microscopically analyzed as amorphous silica and chemically analyzed as 98.9 per cent SiO_2. The seventeenth to twenty-fourth rows of blades contained small amounts of deposit.

TABLE V.—ANALYSES OF MATERIAL REMOVED DURING LOW PRESSURE TURBINE WASHING.

	Dissolved, per cent	Undissolved, per cent
Silica SiO_2	10.9	20.0
Iron and Aluminum Oxides Fe_2O_3—Al_2O_3	0.2	40.0
Sulfate—SO_4	46.2	10.0
Chloride	1.5	
Phosphate PO_4	1.2	

The results obtained after washing and the analyses made show that on the high-pressure turbine, washing was effective and that most of the material deposited on the blades was soluble while on the low pressure turbine, the washing was not very effective and most of the material deposited was insoluble silica. The following questions now arise:

1. Does the turbine deposit indicate carry-over as mechanical entrainment?
2. Does the turbine deposit indicate vaporization?
3. Could it be that vaporization is selective and would have a tendency to remove silica, allowing the positive ion of the salt to combine with the water?
4. Might it be that carry-over from the drum is mechanical and vaporization occurs in the superheater or on the high-pressure turbine blades?
5. It might also be, as Mr. Hall suggests, that vaporization does occur in the boiler at overheated areas or spots of high rates of evaporation at which the salts are vaporized and are not recondensed, but are carried with the steam from the boiler.

The answers to the above questions should materially help in solving the problem now confronting the users of high pressure boilers and turbines.

MR. S. F. WHIRL.[16]—Mr. Morey's paper is a good example of the application of highly theoretical research to an every-day engineering problem. His description of the solvent action of water vapor at high temperature and pressure offers an explanation, other than mechanical carry-over, for the transport of solids from the boiler water into the steam. It also suggests a possible approach to the more important phase of the problem, namely, the selective deposition of solids at various points in the steam system.

Deposits in the superheater result in overheated and ruptured tubes; in the turbine they impair both efficiency and capacity. Consequently, the operator is only casually interested in the mechanism of carry-over and the quantity of solids in the steam. His chief concern is the location and amount of those solids that "stick and stay stuck." For instance, one of our turbines after more than two years of continuous operation was found to be absolutely clean, in spite of the fact that more than a ton and a half of solids accompanied the $10\frac{1}{2}$ billion lb. of steam entering the turbine. It is hoped that the present Edison Electric Institute study of cases such as this one and others of turbine fouling will lead to a better understanding of the problem. Among the factors being considered are the amount and composition of solids, temperature, pressure, velocity, pH of the steam, and the design and operation of the equipment.

The theory of solubility of boiler water solids in steam supplements but does not discredit the role of mechanical carry-over. The former has been suggested as an explanation of the presence of troublesome silica deposits in a number of high-pressure turbines. However, I believe we must rely on the latter to explain such phenomena as the presence of magnetic iron oxide in the steam and the increase in carry-over with rating experienced by most boilers.

MR. W. H. ROWAND.[17] —The discussion has been focused on the assumption that the solids carry-over now being experienced is the result of solubility in the steam. It is my opinion that the ultimate has not yet been reached in reducing carry-over by mechanical means and that this method of attack should still be prosecuted diligently.

The vast number of different solids carry-over tests we have conducted show a narrow band of carry-over ranging from 0.3 to 0.7 ppm.; these tests cover a very wide range of pressures, boiler water concentrations, water levels and ratings. Tests on one boiler operating at 2400 psi. pressure show solids carry-over by the conductivity method of only 0.4 ppm., which is much smaller than that predicted by the solubility theories.

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[16] Chief Chemist, Duquesne Light Co., Pittsburgh, Pa.

[17] Service Dept., The Babcock & Wilcox Co., New York N. Y.

It is quite possible that this relatively small residual carry-over is the result of finely atomized spray or dust which the existing mechanical methods of separation fail to remove from the steam.

I do not wish to belittle the worthwhile progress which has been and will be made by the chemical attack on this problem, but I do want to emphasize that we should continue the mechanical attack concurrently with it.

MR. P. B. PLACE[18] (*by letter*).—The failure to eliminate turbine blade deposits by mechanical purification of steam and the finding of localized and more or less selective deposits on the blades have, without doubt, been the principal incentives in the development of new theories on carry-over.

The concept of carry-over by solubility of boiler water salts in high-pressure saturated steam, followed by selective deposition on turbine blades under suitable conditions of temperature and pressure, is attractive in its possibilities and will receive considerable attention in the near future.

Such a theory, however, suggests that complete elimination of impurity in high-pressure steam may be impossible and that the impurity is likely to be higher as operating pressures increase. Further, it suggests that the progress in mechanical purification of steam, which has paralleled the increase in operating pressure through the intermediate range, cannot be expected to continue.

It is unlikely that perfect separation is possible with the best equipment now available. The normal carry-over from a boiler is in the form of a steam-borne spray. The size of the spray varies from the extremely small particles that make up a thin fog to the largest size droplet that can escape the mechanical separator installed in the drum. The failure to eliminate carry-over completely from high-pressure steam may conceivably be due to the generation of a larger proportion of the finer particles under high-pressure operating conditions.

The selective deposition on turbine blades and localization of such deposits is difficult to explain but the difficulty should not imply that it is impossible. Is it not possible that mechanical forces and the physical condition of the carry-over dust from the superheater have some bearing on the selective deposition? For example, in coal pulverizer exhausters, a very hard deposit is often found which has been built up by accumulation of extremely fine coal dust. It is a selective and localized deposit wholly dependent on mechanical and physical conditions.

In superheaters, deposits are seldom found unless the amount of carry-over is sufficient to wet the metal surface and allow evaporation of a liquid film adjacent to the metal. Small carry-over, as steam-borne spray, evaporates in suspension and the resulting dust is carried through the superheater without deposition. This spray evaporation has an effect on the size and structure of the resulting dust and is a method used in the chemical industry for controlling such products as common table salt. Under various conditions of spray evaporation in superheaters, selective sizing of the individual components of the boiler salines may result with consequent selective deposition on the turbine blades. The beneficial effects of excessive sulfate and the inconsistencies in deposition range in the turbine might be due to variable conditions of spray evaporation in the superheater.

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[18] Combustion Engineering Co., Inc., New York, N. Y.

This discussion is presented, not to refute the solubility theory but rather to suggest a parallel line of thought that may have some bearing on the problem.

COMBINED BIBLIOGRAPHY—ROUND TABLE DISCUSSION ON THE SOLVENT ACTION OF WATER VAPOR AT HIGH TEMPERATURE AND PRESSURE

Since the presentation of this discussion at the annual meeting several pertinent papers have become available and are included in the bibliography. These references appear as references numbers **(38, 44, 46, 51)**.

(1) John Anderson, "Operating Experiences with 1300 lb. Steam Pressure," *Engineering* (London), January 6, 1928, pp. 25–28 January 13, 1928, pp. 55–58; *Journal* Inst. Fuel, Vol. 1, pp. 131–160 (1928).

(2) M. D. Baker, "Mechanical Purification of Steam Within the Boiler Drum," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 61, pp. 711–720 (1939).

(3) H. B. Blumendal, "Vaporization of Silica with Steam," *Recueil des travaux chimiques des Pays-Bas*, Vol. 49, pp. 857–860 (1930); Vol. 51, pp. 708–714 (1932); Vol. 53, p. 476 (1934).

(4) B. O. Buckland, "Turbine Nozzle and Bucket Deposits," *Proceedings*, Second Annual Water Conference, Eng. Soc. Western Pa., pp. 115–130 (1941); "Effect of Nozzle and Bucket Deposits on Turbine Capacity and Efficiency," *General Electric Review*, Vol. 45, No. 2, February, 1942, pp. 123–129.

(5) Perry Cassidy, "The Trend of Boiler Design as Affected by Factors Internal to Boilers," p. 15, paper furnished through courtesy of Babcock & Wilcox printed privately by the company as *Monograph 3-780*, following its presentation before the Engineers Society of Western Pennsylvania, Pittsburgh, October 15, 1935.

(6) N. I. Chitarow and L. A. Iwanow, "Critical Temperature of Water and Aqueous Solutions of Sodium Silicate," *Zentralblatt für Mineralogie*, pp. 46–54 (1936).

(7) Karl Cleve, "The Salt Content of Steam from Steam Boilers," Zeitschrift des Vereines deutscher Ingenieure, Vol. 84, pp. 789–795 (1940).

(8) "Fouling of Turbine Blades," Statement by Texas Power and Light Co., *Proceedings*, Nat. Electric Light Assn., Vol. 88, p. 1017 (1931).

(9) "Fouling of Turbine Buckets," Statement by Duquesne Light Co., *Ibid.*, pp. 1014–1015.

(10) "Fouling of Turbine Buckets," Statement by Southern California Edison Co., Ltd., *Ibid.*, pp. 1015–1016.

(11) W. Geisler, "Silica Removal from Feedwater at the High Pressure Station in Höchst," *Vom Wasser*, Vol. 12, pp. 381–386 (1937). (Washing turbine with silica with hot NaOH. Outlet temperature from 240 to 280 C. no more SiO_2 in the Vorschalt turbine.)

(12) H. Goerke, "Deposits of Salts and Silica in Steam Turbines," Contribution of the Wirtschaftsgrappe Elektrizitätsversorgung, translated by C. F. Stolzenbach, *Electrizitätswirtschaft*, Vol. 25, pp. 614–616 (1939).

(13) R. E. Hall, Discussion of paper by B. O. Buckland, "Turbine Nozzle and Bucket Deposits," *Proceedings*, Second Annual Water Conference, Eng. Soc. Western Pa., pp. 125–127 (1941).

(14) R. E. Hall, "Water Conditioning for High Pressure Steam Generation," *Proceedings*, Nat. District Heating Assn., Vol. 27, pp. 232–252 (1936).

(15) *Journal*, Chem. Soc. (London), Vol. 38, p. 210 (1880).

(16) N. B. Keevil, "Vapor Pressures of Aqueous Solutions at High Temperatures," *Journal*, Am. Chemical Soc., Vol. 64, No. 4, pp. 841–850 (1942).

(17) A. Kleinhans, "Vaporization of Boiler Salts—The Part of Vaporized Salts in the Salt Content of Steam," *Archiv für Wärmewirtschaft und Dampfkesselwesen*, Vol. 17, pp. 127–128 (1936).

(18) B. Koch, "Volatilization of Boiler Salts," *Die Wärme*, Vol. 61, pp. 219–223 (1938).

(19) F. C. Kracek, "*P-T-X* Relations for Systems of Two or More Components and Containing Two or More Phases (L-V, L_I-L_{II}-V and S-L-V Systems)," International Critical Tables, Vol. III, p. 370 (1928).

(20) F. C. Kracek, "The System Na_2O-SiO_2," *Journal of Physical Chemistry*, Vol. 34, p. 1583 (1930).

(21) Ernst Lupberger, "Newer Development of Steam Boiler Building," *Stahl und Eisen*, Vol. 53, pp. 1080–1084 (1933).

(22) A. A. Markson, "Natural Circulation Problems," *Proceedings*, Midwest Power Conference, Vol. 5, pp. 188–1 6 (1942).

(23) J. W. Mellor, "Modern Inorganic Chemistry," Longmans, Green and Co., London (1930).

(24) F. Michel, Discussion of paper by F. G. Straub, "The Cause and Prevention of Steam Turbine Deposits," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 58, pp. 330-331 (1936).

(25) G. W. Morey, "New Crystalline Silicates of Potassium and Sodium, Their Preparation and General Properties," *Journal*, Am. Chemical Soc., Vol. 36, pp. 215–230 (1914).

(26) G. W. Morey, "The Volatility of Silica with Steam," *Transactions*, Am. Geophysical Union, Thirteenth Annual Meeting, pp. 269–270 (1932).

(27) G. W. Morey and C. N. Fenner, "The Ternary System H_2O-K_2SiO_3-SiO_2," *Journal*, Am. Chemical Soc., Vol. 39, pp. 1173–1229 (1917).

(28) G. W. Morey and Earl Ingerson, "The Pneumatolytic and Hydrothermal Alteration and Synthesis of Silicates," *Economic Geology*, Supplement to Vol. 32, No. 5, August, 1937, pp. 607-761.

(29) G. W. Morey and Earl Ingerson, "The System, Water-Sodium Disilicate," *American Journal of Science*," V, Vol. 35-A, pp. 217–225 (1938).

(30) A. R. Mumford, "Studies of Moisture at High Rates of Evaporation," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 51, pp. 363–370 (1929).

(31) F. R. Owens, Discussion of paper by B. O. Buckland, "Turbine Nozzle and Bucket Deposits," *Proceedings*, Second Annual Water Conference, Eng. Soc. Western Pa. pp. 124–125 (1941).

(32) E. P. Partridge, "Formation and Properties of Boiler Scale," *Engineering Research Bulletin No. 15*, pp. 67–84, University of Michigan (1930).

(33) Power Stations Chemistry (1937) Turbine Blade Deposits, *Publication No. E-10*, September 1937, pp. 5–7; *Publication No. H-11*, Edison Electric Inst., July, 1941, pp. 14–17.

(34) H. M. Rivers and W. P. Hill, "The Elimination of Carryover Under Steel Mill Operating Conditions," *Transactions*, Am. Soc. Mechanical Engrs. (1943).

(35) W. C. Schroeder, A. A. Berk, and A. Gabriel, "Solubility Equilibria of Sodium Sulphate at Temperatures from 150–350°. 2. Effect of Sodium Hydroxide and Sodium Carbonate," *Journal*, Am. Chemical Soc., Vol. 58, pp. 843–849 (1936); "Effect of Sodium Hydroxide and Sodium Phosphate," Vol. 59, pp. 1783–1790 (1937).

(36) W. C. Schroeder, Alton Gabriel, and Everett P. Partridge, "Solubility Equilibria of Sodium Sulphate at Temperatures from 150–350°. 1. Effect of Sodium Hydroxide and Sodium Chloride," *Journal*, Am. Chemical Soc., Vol. 57, pp. 1539–1546 (1935).

(37) Erich Schröer, "The Critical State of Water and Aqueous Solutions," *Zeitschrift für physikalische Chemie*, Vol. 129, pp. 79–110 (1927).

(38) M. C. Schwartz, "Critical Problems in the Boiler Feed Water Field," *Journal*, Am. Water Works Assn., No. 11, pp. 1214–1226 (1942).

(39) M. C. Schwartz, W. B. Gurney and T. E. Crossan, "Determination of the Purity of Steam by Gravimetric and Spectrographic Methods," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 62, No. 8, November, 1940, pp. 720–721 (Tables 1 and 2).

(40) A. Smits, "Über die Erscheinungen, welche auftreten, wenn bei binären Gemischen die Faltenpunktskurve der Löslichkeitskurve begegnet," *Zeitschrift für physikalische Chemie*, Vol. 52, pp. 587–601 (1905).

(41) A. Smits, "The System Water-Silicon Dioxide," *Recueil des travaux chimiques des Pays-Bas*, Vol. 49, pp. 962–966 (1930).

(42) C. R. Soderberg, Discussion of paper by F. G. Straub, "The Cause and Prevention of Steam Turbine Deposits," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 58, pp. 329–330 (1936).

(43) F. Spillner, "High Pressure Steam as a Solvent—the Cause of Salt Carry-Over by Steam in High Pressure Boiler Plants," *Die Chemische Fabrik*, Vol. 13, No. 22, November, 1940, pp. 405–424.

(44) A. Splittgerber, "The Behavior of Boiler Water Salts in Superheater and Turbine," *Vom Wasser*, Vol. 12, pp. 366–380 (1937).

(45) A. Splittgerber, see reference (44), pp. 375–380.

(46) A. Splittgerber, "The Chemical Characteristics of Silicic Acids Dissolved in Water, and Their Influence on High Pressure

Boiler Operation," *Vom Wasser*, Vol. 15, pp. 271–332 (1940).

(47) A. Splittgerber, see reference (46), pp. 300–302.

(48) A. Splittgerber, "Concerning the Formation of Salt and Silicate Deposits in Superheaters and Turbines, and Corrective Measures," *Technischen Mitteilungen* des Hauses der Technik, Essen, Vol. 31, p. 597 (1938); *Vom Wasser*, Vol. 15, p. 296 (1940).

(49) "Steam Engineering Abroad (as Reported in the Foreign Technical Press)," *Combustion*, Vol. 13, No. 7, p. 47 (1942).

(49a) F. G. Straub, "Analcite-Preparation and Solubility Between 182 and 282 C.," *Industrial and Engineering Chemistry*, Vol. 28, p. 113 (1936).

(50) F. G. Straub, "The Cause and Prevention of Steam Turbine Deposits," *Bulletin No. 282*, pp. 11–13, Engineering Experiment Station, University of Illinois (1936); *Transactions*, Am. Soc. Mechanical Engrs. Vol. 57, pp. 447–454 (1935).

(51) F. G. Straub, "Solubility of Salts in High Pressure Steam," abstracted in *Combustion*, Vol. 14, No. 5, November, 1942, pp. 51–52. Paper presented at Third Annual Water Conference, under auspices of Engineering Soc. of Western Pa. Paper and discussion will be available in 1943.

(52) Symposium on Problems and Practice in Determining Steam Purity by Conductivity Methods, *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 1261 (1941).

(53) S. E. Tray, "Boiler Operation as It Affects Prime Movers," *Mechanical Engineering*, Vol. 60, pp. 475–480 (1938).

(54) "Turbine Blade Deposits," *Report H-11*, Edison Electric Inst., July, 1941.

(55) C. J. van Nieuwenburg and P. M. van Zon, "Semi-quantitative Measurements of the Solubility of Quartz in Super-Critical Steam," *Recueil des travaux chimiques des Pays-Bas*, Vol. 54, pp. 129–132 (1935).

(56) M. Werner, French Patent 802,429 for I. G. Farbenindustrie A.-G., Ausgegeben September 4, 1936, with *Wirkung* of February 26, 1936, German priority February 26 to July 23, 1935; *Chemisches Zentralblatt*, Vol. II, p. 3829 (1936).

(57) M. Werner, "The Smooth Ice and Hoar Frost Theory of Turbine Deposits," Patent application I. 51,621, Kl. 85bl/01 applied for February 7, 1935; Ausgelegt August 12, 1937; Withdrawn June 9, 1938.

(58) W. Wesley and W. Geisler, "Experiences Concerning the Feeding of High Pressure Boilers with Chemically Prepared Water," *Vom Wasser*, Vol. 12, pp. 319–340 (1937).

SYMPOSIUM ON RADIOGRAPHY

SOME APPLICATIONS OF X-RAY INSPECTION TO PRODUCTION PROBLEMS

By Don M. McCutcheon[1]

The problem of applying X-ray inspection to the manifold phases of War Production is of considerable general interest, and may be ascribed to two specific causes. One is the high-volume production of cast light alloys and other metals required for aircraft construction. The second is the extension of high-strength steel castings into new fields formerly dominated by forgings. Special applications and Government specifications probably also account for a considerable extension of X-ray inspection in some fields.

X-ray inspection is expensive and has in general been previously applied by the cast metals industry to the development of a proper foundry technique. The designing engineer usually has allowed a generous factor of safety in the design which is sufficient to take care of any minor defects which arise from conditions usually beyond the control of the average economical foundry technique. The use of cast parts in moderately stressed sections and the limitation on weight imposed by certain applications has necessitated a proper balance between foundry methods that are economically possible from a production standpoint, and the individual testing by X-ray inspection.

Another problem confronting the metallurgical engineer is the substitution of cast parts for forgings as a result of the present limited forging volume available. The parts may or may not be redesigned as circumstances or applications permit. In either case soundness of castings is a requirement which must be considered. Occasionally the cast part used must have equal strength with the equivalent forging, but generilly specifications limit the load to much lower values. Individual cases may determine the extent to which X-ray inspection must be applied.

The application of X-ray inspection to cast production parts is dictated largely by the design and application of the part. Very often a part can either be designed with an adequate safety factor or it may contain sections of low stress which allow the development of a foundry technique which insures that any unsoundness which may develop is confined to these low-stress areas. In this case X-ray inspection may only be required during the development of a proper foundry method. Cast metal parts used in highly stressed areas and whose design or weight is severely restricted generally require 100 per cent X-ray inspection. The great majority of cast parts made today may be classified as coming somewhere between these two groups. A special class of casting made by the centrifugal process is receiving increasing attention in armament manufacture. Centrifugal castings, inspected by X-ray and magnetic methods, are being used for very highly stressed parts with distinct advantages from a manufacturing standpoint and often with superior performance.

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[1] Metallurgical Engineer, Main Laboratory, Rouge Plant, Ford Motor Co., Dearborn, Mich.

There are several methods which may be worked out on the extent of the X-ray inspection to be applied. One involves the development of a casting-inspection method based upon the percentage of defective castings found by 100 per cent X-ray examinations for a definite period. This serves mainly as a control of the manufacturing process, and indicates the trend of quality. Adequate inspection by this procedure can only be secured if the volume of identical castings involved is large enough to apply sampling methods. Another method which is used extensively is to base the percentage of castings to be X-ray inspected on the ratio of the design load to material strength. In other words, the design factor of safety dictates the percentage of castings in any one lot which are to be X-ray inspected. The classification of cast parts according to the design load may be made very simple if the foundry process and control is very good. The trend, however, will be to more elaborate specifications and classifications which will allow a greater flexibility of acceptance standards. A reduction in rejections of doubtful cases would be expected. In any case, the correct procedure should be based upon actual breakdown tests upon individual parts. The function of X-ray inspection is to determine the suitability of a part for the purpose for which it is designed. The X-ray equipment used, the methods of application, and the interpretation of

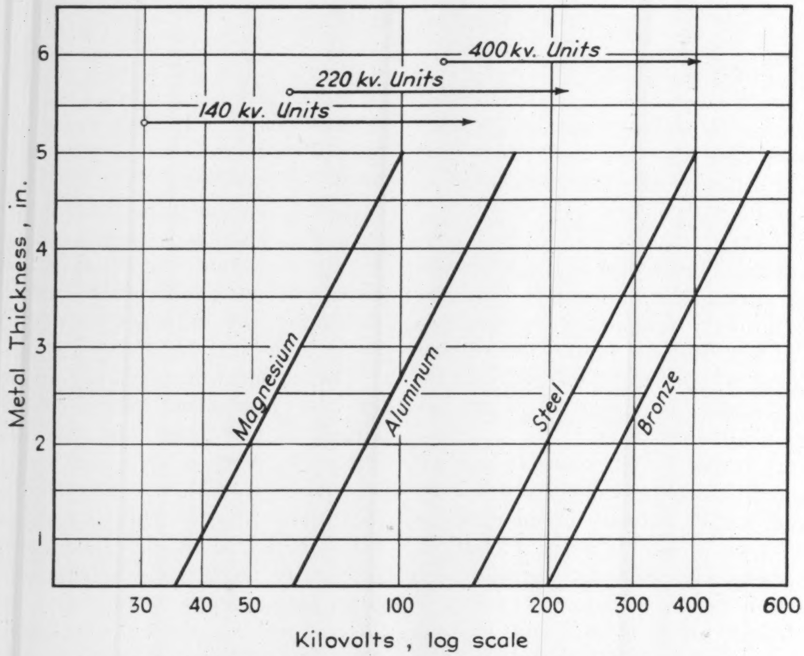

FIG. 1.—Ideal Voltage Range Suitable for Various Metals.

This graph shows the approximate voltage ranges applicable to various materials with the methods available to the radiographer and is intended only as a guide in the selection of equipment.

results are all subject to this main purpose.

Specific Applications of Radiographic Examination

Cast metals in common use today are grouped rather closely in two divisions in the periodic systems of the elements. The light metals and alloys such as aluminum and magnesium have X-ray-absorption values which require the use of low-voltage equipment in order to secure the maximum differential of absorption with thickness. Steel, brass, and other higher atomic weight metals require that a more penetrating radiation be used.

The ideal voltage range suitable for the various metals is shown in Fig. 1. At the top of the chart is indicated the useful voltage range of the common commercial X-ray units available today. The choice of equipment depends upon the metal to be examined and the average section thickness of the parts. In general it can be seen that the 200 to 250-kv. units are the most universal in range and application. For the examination of the light metals magnesium and aluminum, the 140-kv. unit gives the highest efficiency. Likewise, where heavy steel, brass, or bronze castings are to be examined the 400-kv. unit is applicable. Recently, higher-voltage X-ray units have been designed for commercial use and will be discussed later.

Light Metals and Alloys:

The equipment and technique suitable for the X-ray examination of aluminum and magnesium alloys requires the careful consideration of a number of factors not commonly encountered in the X-ray examination of the more familiar cast steel parts. Low X-ray absorption is a characteristic of the light alloys which must be compensated for by the use of low-voltage, or long-wave-length X-ray. The type of defects encountered, their size and distribution, requires that a reasonable degree of image sharpness be obtained. Sharpness of radiographic detail is essentially governed by the distance from the object to the X-ray tube and by the size of the focal spot of the X-ray tube. Some compromise must be reached on the limiting size of focal spot, and the distance from the object to the tube in order to obtain reasonable exposure times. Most X-ray units commercially available in the low-voltage range have focal spot sizes that are sufficient for all general casting examination.

Small light-alloy castings with relatively thin sections requiring long-wave-length X-rays places a limitation on the use of the higher-voltage X-ray units due to their increased inherent filtration. The glass portion of the X-ray tube, the insulating oil, and its bakelite case all contribute to the absorption of the required low-voltage X-rays. X-ray units of 150-kv. capacity or lower are designed with sufficiently thin glass parts on the X-ray tube and thin enclosing structures to allow efficient X-ray output at 30 kv. or less. The more universal 220-kv. unit has a higher practical low-voltage limit which places a restriction on the radiographic detail obtainable. With careful film selection and interpretation this factor may not be objectionable with the average light-alloy sections.

The procedure best followed in radiographing light-alloy castings is to use the lowest voltage on the X-ray tube consistent with a reasonable exposure time. Where a large number of castings are X-rayed per day, the most convenient technique is to establish a reasonable exposure time, and select the voltage required for the section thickness being X-rayed. No exposure should be made for less than 1 min. with low-milliamperage X-ray units. This insures that the radiographic operator uses the lowest voltage practical with the thinner metal sections. An additional requirement is made that all films show the penetrameter gage designed for the section thickness being X-rayed. For thin aluminum and magnesium alloy sections satisfactory penetrameter sensitivity is difficult to obtain unless low voltage radiation is used and correct developing technique followed. Fine-grained, high-contrast X-ray films are necessary for resolving the small holes in these thinner gages. These high-contrast, fine-grained films are particularly applicable where the X-ray unit used has limited low voltage range or above-normal inherent X-ray filtration.

The successful examination of complex shaped castings of aluminum and magnesium alloys requires considerable care in selecting the proper angles and position of casting with respect to the direction of the X-ray beam. In order to minimize the number of exposures required with complex casting shapes, a long tube-to-object distance is recommended. The casting angles used may place some sections of the part several inches off the film. This requires that the ratio of the distance from the X-ray tube to the object and the distance from the object to the film be such that adequate sharpness of detail will be obtained. This factor of detail sharpness is a property of the X-ray unit used and must be individually determined, as will be discussed later.

Where it is not possible to register

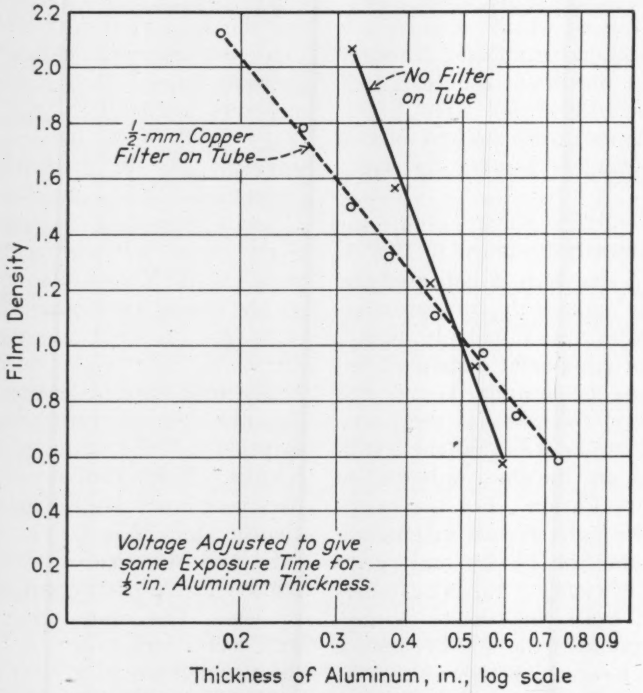

FIG. 2.—Effect of Filtration on the Primary X-ray Beam.

on the film all the casting sections within an acceptable density range, filtered radiation may be used. A metal of higher atomic number than the part being X-rayed is placed on the tube port and the voltage on the unit raised to give the same exposure time used with the unfiltered beam. A special marker is placed on the film indicating the filter used which aids in the proper interpretation of the film.

The effect of filtration on the primary X-ray beam is shown in Fig. 2, where copper is used as a primary beam filter for the examination of aluminum alloys. A limitation on the amount of filtration which may be employed with most structural castings is established by the requirement that a gage sensitivity of 2 per cent of the section thickness be obtained on the film. Filtration of the X-ray beam in any case must not be so excessive that portions of the film exposed to the direct X-ray beam will transmit any of the light being used for viewing purposes. A considerable loss in available film contrast will result from such excessive filtration.

The generally accepted opinion that X-ray beam filtration always results in a loss of sensitivity can be shown to be misleading. In aluminum alloy aircraft castings there are two exceptions to this rule and an increased sensitivity can be shown by the use of proper filtration of the beam. Some aluminum alloy castings, particularly those having a large macro grain structure and adjacent thick and thin sections, will show a striated type of scattering often mistaken as defects in the metal. Removal of the long-wave-length portion of the beam by proper filtration will largely eliminate or reduce this effect. Figures 3 and 4 show the effect of filtration on this type of scattering in an aluminum alloy casting. Another exception to the belief that filtration always results in loss of sensitivity is found with complex shaped castings where large amounts of radiation scattered from adjacent surfaces can reach the film unimpeded by any section of the casting itself.

TABLE I.—RECOMMENDED X-RAY FILTRATION.

Kilovoltage	Application	Section Thickness, in.	Filter
15 to 60...	Magnesium alloys	⅛ to 4	No filter at tube or film.
30 to 60...	Aluminum alloys	⅛ to 1	No filter at film, copper ½ mm. thick at tube for special sections, optional.
60 to 100..		½ to 2	No filter for plain sections, ½ mm. copper at tube for complex shapes. 0.002 to 0.005 in. copper foil at film, optional.
140.........		Above 2	Copper or lead filter 0.005 in. thick at film surface.
125 to 250..	Steel	⅛ to 1	Lead filter at film surface 0.005 in. thick. Lead filter at tube 0.010 in. optional.
220 to 400..		1 to 2½	Lead filter 0.005 to 0.025 in. thick at film. Lead filter 0.010 in. tube optional.
1000........		2½ to 5	Lead filter 0.005 to 0.250 at film depending upon irregularity of section. Lead filter up to ½ in. at tube for round and irregular sections.

Removal of scattered radiation, which is produced by the long-wave-length component of the X-ray beam striking adjacent surfaces of a complex casting, can be secured by use of a metal filter at the tube port or between the object and the film. The location of the filter can best be determined by the X-ray operator and will depend upon the convenience and effectiveness of either method. General scattered radiation, produced by the emerging X-ray beam from surfaces of the casting not in contact with the X-ray film holder, can best be reduced by use of a metal filter in contact with the front or top surface

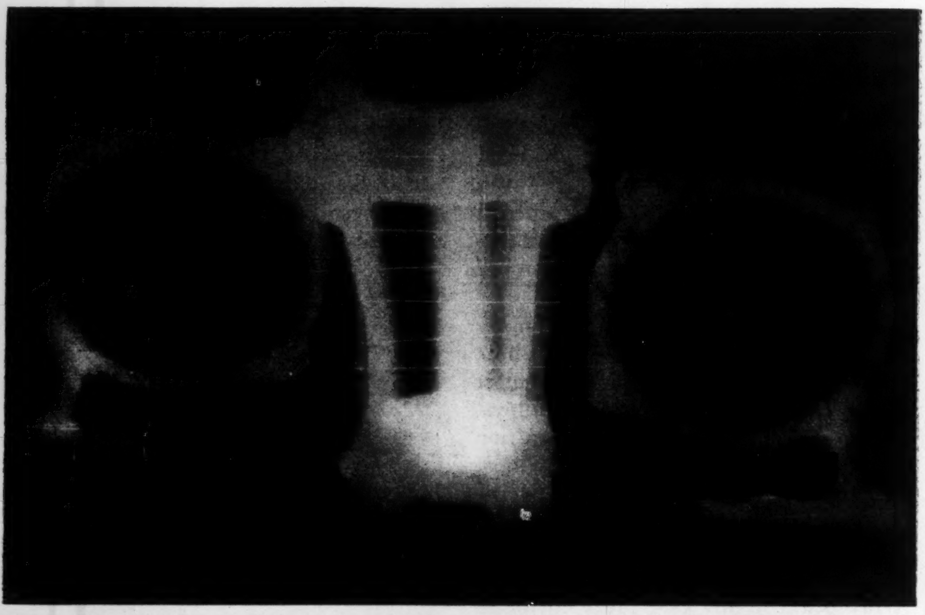

FIG. 3.—Excessive Scattered Radiation with Aluminum Alloy Casting Using Standard Radiographic Method. Tungsten wire grid used to determine the penetration limits of the X-ray exposure.

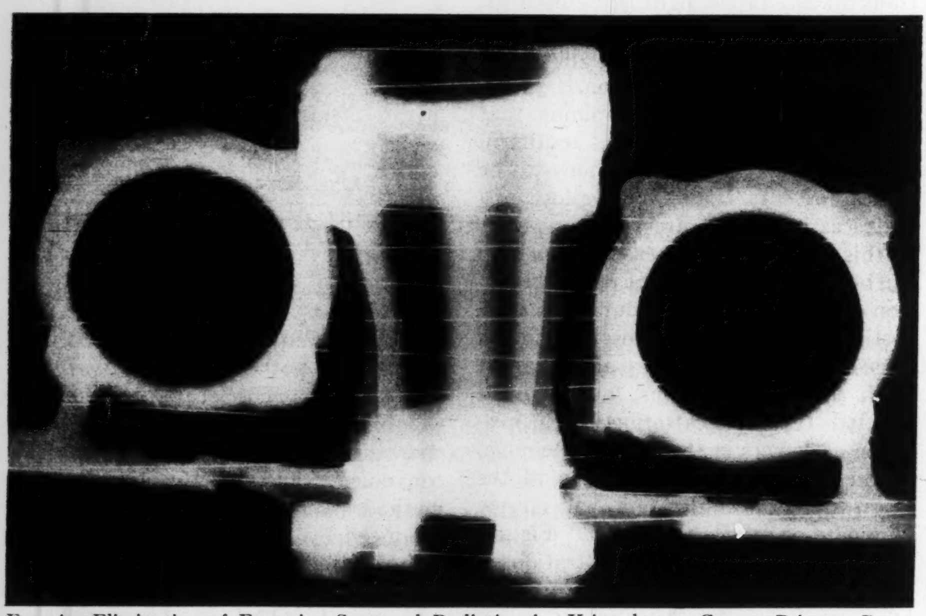

FIG. 4.—Elimination of Excessive Scattered Radiation by Using $\frac{1}{2}$-mm. Copper Primary Beam Filter. Tungsten wire grid indicates penetration limits and absence of scattered radiation.

of the film. Table I lists the most commonly used filter thickness and application used by The Ford Motor Co. Laboratories.

Steel and Other Alloys:

The X-ray examination of steel and copper alloys requires high X-ray intensity. The design of X-ray tubes for high energy output requires larger focal areas on the target and a considerable increase in the thickness of the glass envelope of the tube to withstand the higher electrostatic tension produced on the glass by high-voltage fields. A natural consequence of these factors is a decrease in the sharpness of detail obtainable on the film, and an increase in the inherent filtration of the tube. Figure 1 shows the voltage range that can be used to produce a useful X-ray output on X-ray tubes commercially available today. The recent development of beryllium metal windows for X-ray tubes may have an important bearing on the adaptability of future tubes.

One of the main problems encountered with the use of X-ray beams generated by voltage between 100 kv. and 700 kv. is scattered radiation. Above approximately 700 kv. the scattered radiation produced is largely in the forward direction which simplifies the radiographic technique remarkably. Control of scattered radiation is based upon restricting the area of the primary beam to the object being examined and upon the use of filtration. For relatively thin sections and moderate voltages, filters interposed between the object and the film are sufficient to both restrict the intensity of longer wave length components in the beam from directly striking the film, and to markedly reduce the scattered X-ray intensity emitted by all objects in the path of the primary rays. For irregular-shaped castings a double filter technique has been found advantageous.[2] The double filter technique listed in Table I requires the use of lead filters both on the X-ray tube port and between the object and the film. A higher X-ray tube voltage is usually required when using the double filter to secure a reasonable exposure time, but the increase in film clarity obtained with complicated castings, by a greater reduction of scattered rays, more than offsets any loss in contrast which theoretically results from the use of higher voltages. Full use of the double-filter technique for irregular-shaped steel castings is limited to the following maximum section thickness on the X-ray units commercially available:

220 kv.	1¼ in. of steel
400 kv.	2½ in. of steel
1000 kv.	6 in. of steel

The thickness of metal which may be examined by X-rays between 150 kv. and 400 kv. may be extended by the use of calcium tungstate intensifying screens to the following maximum limits with reasonable exposure time:

150 kv.	1 in. of steel
220 kv.	3½ in. of steel
400 kv.	5 in. of steel

Lead foil filters should always be used between the object being X-rayed and the front intensifying screen to reduce the effect of the scattered radiation.

The principal disadvantage in the use of intensifying screens, aside from image diffusion, is that in all cases the direct primary beam must be prevented from striking the film holder. This

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[2] H. E. Seemann, "Secondary Radiation in the Radiography of Aluminum, Steel, and Lead," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part II, p. 284 (1938); "Secondary Radiation Intensity as a Function of Certain Geometrical Variables," *American Journal of Roentgenology and Radium Therapy*, Vol. XXXIV, No. 4, April, 1938; "Some Physical and Radiographic Properties of Metallic Intensifying Screens," *Journal of Applied Physics*, Vol. 8, No. 12, December, 1937.

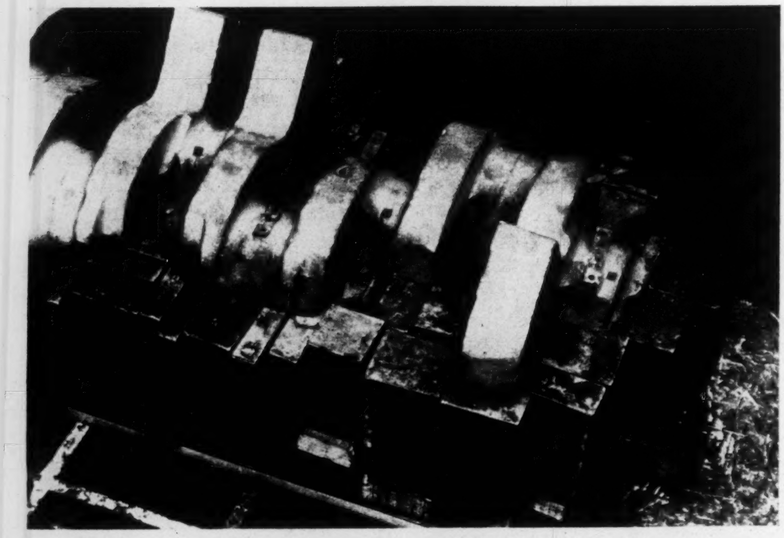

FIG. 5.—Blocking Method Required to Obtain a Good Radiograph with Calcium Tungstate Intensifying Screens at 400 kv.

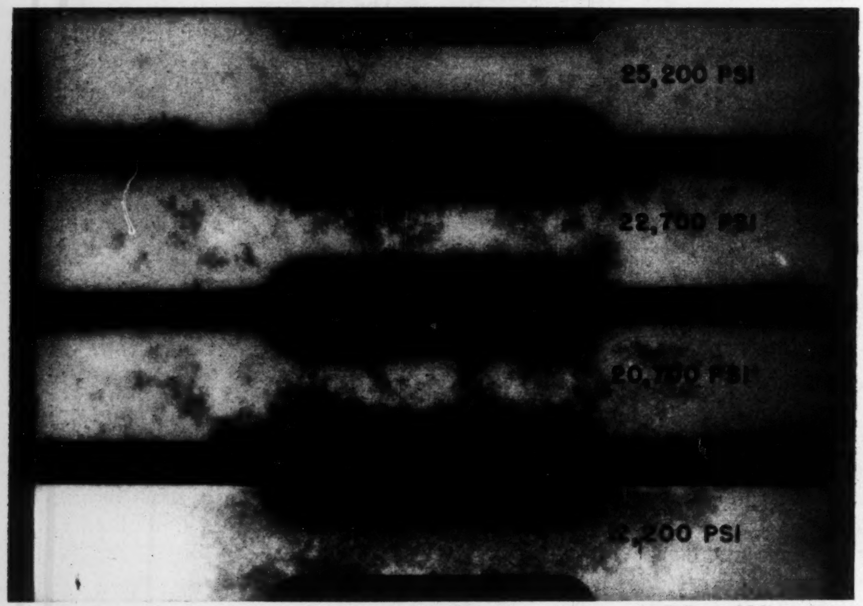
25,200 PSI
22,700 PSI
20,700 PSI
12,200 PSI

FIG. 6.—Radiographs of Machined Magnesium Alloy Test Bars Showing the Effect of Microshrinkage on Tensile Strength.

requires the very careful and time-consuming "blocking" methods of all irregular shaped castings which do not completely cover the film holder. Any high absorbing material, such as lead shot, steel blocks, or radiographic putty may be used to fill in those areas on the film holder which are open to the direct primary beam. Figure 5 shows the blocking method required for the X-ray examination of a cast crankshaft, where a combination of heavy steel blocks is used in conjunction with radiographic putty.

The necessity for using intensifying screens and the troublesome and time-consuming blocking technique for heavy sections is eliminated by the use of the million-volt X-ray unit which will be described later. Before leaving the discussion of low- and medium-voltage X-ray units, their adaptation to high-volume casting examination and special applications in new fields will be reviewed.

Automatic X-ray Units:

Aircraft construction requires the use of a large quantity of small aluminum and magnesium castings. A considerable number of these castings are subjected to appreciable stress, and consideration must be given to their soundness and freedom from defects in critical sections. The effect of microshrinkage on the tensile strength of machined magnesium alloy bars is shown in Fig. 6.[3] The use of X-ray examination as an invaluable aid in the development of a foundry technique for producing sound castings has been previously mentioned. X-ray examination must now serve as a very prominent and vigilant control of the entire casting process. As X-ray examination is an expensive control process, agreement on the sampling methods must be made between the Government agency and the producing or consuming parties. Even after establishing a percentage control basis for X-ray examination, large aircraft construction projects require that many thousands of parts be examined per day.

The change from the conventional batch-type methods of X-ray examinations to a straight-line conveyor type mechanism with the X-ray unit in the center is a natural development of the mass-production trend. Straight-line motion of parts on a conveyor belt or tray arrangement requires the placing of the X-ray tube in a lead-lined tower arranged to receive the parts automatically, carry out the X-ray operation, and discharge the parts on the opposite side. The mechanical design of such a unit may be constructed in a number of ways according to the same basic principle of semicontinuous operation.

A large variety of size and shapes of cast aluminum, magnesium, and steel are used in aircraft construction which makes it difficult to use one X-ray tube for all parts. Fortunately where the volume of castings being examined by X-rays is large, as at the Ford Willow Run Plant, more than one automatic X-ray unit may be used allowing the selection of tubes specially suited for the class of work involved.

The general progress of castings through a production X-ray department will be according to the following plan. Castings as received will be segregated into groups according to the X-ray unit best adapted for their examination. Sorting tables, numbering machines, and record entries precede the transfer to gravity conveyors where operators place the castings on the film cassettes with proper fixtures and identification numbers. The assembled group is manually pushed onto the conveyor belt which automatically carries the group into the X-ray zone and out the opposite side according to a set cycle of operations. Disassembly of the castings, and storage of the parts in numbered bins follows until final disposition can be made after inspection of the X-ray films.

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[3] The effect of defects on performance of actual castings in service is usually less than this test indicates. However, the type and location of the defect should be considered in relation to the amount and kind of stresses involved.

The processing of X-ray films in the dark room must be at a rate sufficient to keep up with the automatic X-ray machines. This involves equipment which requires a minimum of handling and of sufficient capacity to hold all the films received in the period from the beginning of development to dry films outside the dark room. An automatic developing machine should be used to process all films during peak production. A batch-type developing unit may be added as a supplement to the automatic unit for off-peak loads and in the event of any mechanical failure. Film drying in the automatic developing unit is accomplished by conveying the washed films through an infrared drying zone where large volumes of warm air are passed over the wet films to facilitate the removal of the water vapor.

Considerable care must be given to the convenient preparation of the chemical solutions used for film processing and adequate film and chemical storage space. Adjacent to one end of the dark room may be placed an air-conditioned room for the storage space and chemical solution makeup. Films and chemicals will be stored on the ground floor level, but the solution makeup tanks may be located on the second floor to facilitate the solution transfer by gravity feeding to the dark room processing tank.

The volume of castings examined on the automatic X-ray machines requires a degree of control in all phases of the operation beyond that normally encountered in X-ray methods. Automatic primary voltage regulator should be used, for example, on each X-ray machine to maintain a more constant voltage on the X-ray tube. Film processing control is based upon tests and rules which, although well known, are not generally adhered to in the dark room.

Close control of developing conditions may be made by following closely the advice of the major film producers or actual routine tests may be established in the individual laboratory. It is common practice in dark-room procedure to count the films passing through the solutions, and with adequate additions of developer replenisher a reasonable degree of control can be maintained. Use of a good densitometer for determining actual film density is recommended.

The procedure in one laboratory which has shown considerable promise is to expose a film to a standard amount of ordinary light intensity and measure the film density after processing. Information will be obtained on the strength of the developer, amount of chemical fog, and time required to clear the film in the fixer. The establishment of definite rules and control of the processing solutions will result in the maintenance of consistent, high-quality, radiographic films necessary for production X-ray work.

Million-Volt X-rays:

Radiographic examination of cast metals received considerable impetus with the introduction of million-volt X-ray units. The limitations of lower-powered X-ray units on thickness penetration and the very troublesome scattered radiation were offset to a remarkable degree by the use of the increased voltage.

The design of building facilities for the million-volt X-ray units involves large structures and considerable expense. Where the size of the work, and principally the volume of work, is large the million-volt unit is an economical investment. On the other hand, where the volume of work is small it would appear that radium would be more economical for heavy castings. As a basis of comparison on the one part, the million-volt unit can examine 64 times the volume formerly X-rayed on the 400-kv. unit and 24 times the volume possible with radium of average amount in the same floor area and with the same handling facilities. The difficulty with making comparisons is that it does not allow a fair comparision of the maximum operating efficiency for each method. Thus, the lower-powered units are still the best investment within the limits of the size of sections ideal for their voltage range.

The efficiency of the million-volt X-ray unit is exceptionally high for two reasons: First, the great reduction in scattered radiation produced which allows the X-ray examination of most castings without using any blocking technique. The object is simply placed in front of the film holder and exposed. Second, the utilization of as much as possible of the spherical beam given off by this unit. In low-voltage X-ray units the X-ray tube target is approximately 20 deg. off the normal to the tube axis, and the focal area on the target is rectangular in shape. In order to secure a reasonably uniform focal spot as seen from the film the beam angle must be restricted to rather narrow limits. This limits the useful X-ray zone and restricts the volume which can be examined at each exposure.

The tungsten target of the million-volt unit is very thin and causes little absorption of the X-ray beam except near the plane of the target. X-rays emanating from the lower hemispherical area are called the transmitted rays, and those emanating in the available region above the target are called the reflected rays. As the target focal spot is very small and circular, the apparent size from any angle is approximately the same. Due to the large spherical angles available for useful radiographic examination a large number of castings can be examined at each exposure. The limiting factor on the number of castings that can be examined each day is almost entirely dependent upon the handling facilities provided. At the Ford Motor Co., as many as 50 films have been simultaneously exposed.

There are two methods in use for providing proper protective and working structures to house the million-volt X-ray unit. In the welding field, a welded boiler for example may be located below ground level in a concrete pit and circumferential and longitudinal welded seams X-rayed without causing excessive radiation to reach the floor level. For foundry inspection of heavy steel castings, the X-ray unit is most conveniently located in a heavy concrete-walled room. The working space provided and available handling equipment largely determines the usable efficiency of the million-volt unit.

Operation of the million-volt unit does not differ in essential details from conventional lower-voltage practice. The use of heavy lead filters both at the tube and at the film is common practice for most of the casting work encountered. For flat sections, such as armor plate, it is not necessary to use heavy filtration, the 0.010 to 0.050-in. thick lead foil filters adjacent to the film being sufficient. At 1,000,000 v., for example, the absorption of $\frac{1}{2}$ in. of lead is equivalent to only 3 in. of steel. For very irregularly shaped castings experience has shown that best results will be obtained by using $\frac{1}{8}$ in. or more lead sheet filter in front of the X-ray film. The specific type of work being examined determines whether any filtration should be added to the tube in addition to the lead at the film. The principle of the double-lead filtration technique previously discussed for lower-voltage units applies equally well for the million-volt X-rays.

Examination of large numbers of castings per day on the million-volt unit, as previously noted, is facilitated by utilizing as much of the spherical beam given off by the X-ray unit as possible. Fixtures, racks, cranes, and carts are all necessary accessories found indispensable for rapid handling of heavy parts. Where possible all preparation and adjustments of position are made in a preparation room adjacent to the X-ray room. Time consumed in the X-ray room proper should be kept at a minimum to reduce the time lost between exposures.

The second million-volt installation at the Ford Motor Co. is designed on the same principle as the automatic-type X-ray units described in a previous section. Two large preparation rooms are built at each end of the concrete X-ray room with a standard-gage railway track running through all three rooms. Two flat cars are used to carry a large number of prepared castings alternately into the X-ray room for immediate exposure. During each exposure the second flat car is being reloaded with castings and the necessary films and markers placed in position. The most efficient utilization of the million-volt X-ray unit is possible by this method. All three rooms are provided with overhead electrically operated heavy-duty cranes to provide rapid handling of heavy castings.

Large castings requiring more than one film are marked off into sections according to a standard sketch of the part on file in the control room. Technique required for each section is logged on the standard sketch, and the preparation room crew arranges the details and positions the film holders on the castings before moving them into the X-ray room. Frequently the technique can be arranged so that all of the casting, regardless of section thickness, can be taken at one exposure. For example, the examination of 3 in. of steel adjacent to a 1.0-in. section can be made by placing two film holders back of the casting. The total lead filtration thickness before reaching the last film is regulated to compensate for the difference in casting thickness.

A heavy lead diaphragm is used on the million-volt unit to eliminate any radiation which emanates from other than the principal target area. Electrons which reach the target and are not completely absorbed will rebound and produce off-focus radiation which is very penetrating. Considerable improvement in film clarity and image sharpness is attained by using a long tube-to-object distance and a well-diaphragmed beam.

The construction of the General Electric million-volt X-ray unit does not allow a wide range of usable kilovoltage. This restriction tends to limit the use of the unit to steel sections under 1.0 in. thick if good sensitivity must be maintained. Considerable improvement in contrast and sensitivity may be obtained on steel sections from 0.5 to 3.0 in. if the reflected beam, making approximately 120 deg. with the direction of the electrons, is used instead of the direct or zero-degree beam. Absorption tests have shown that the reflected rays have a longer effective wave length than the direct or zero-degree rays.

Experience has shown that a higher average film density is required with million-volt radiography than is customary with lower voltage technique. An average film density of 2.0 is suitable with the newer type of viewing boxes while higher film densities obtained as a result of wide variation of section thickness can readily be examined by using high-powered "Keg" type spot lights suitably filtered to remove the heat rays. Fluorescent-type viewing boxes containing eleven lamps are commercially available and are ideal for these high average film densities.

Million-volt radiography applied to the heavier cast and welded steel sections has made possible a wider application of the stereoscopic method for viewing the section in three dimensions. As an aid in determining the necessity for repair welding and for accurately locating the position of the defect, stereoscopic technique is being used with more success than was possible on the thinner metal sections. The appearance of fine cracks in a 2-in. welded armor section were successfully correlated with the particular welding pass and the depth measured which materially aided in the repair of the section. Heavy castings containing mild shrinkage areas may often be scrapped if subsequent machining runs into the defect. Stereoscopic radiography can generally determine this possibility thereby saving both the time and expense of machining. Repair welding of local shrinkage areas in line with machined surfaces may often be possible only on the rough casting due to the restriction on welding and heat treating after machining operations. The technique of stereoscopic radiography is discussed in later sections of this Symposium.

Use of Radium:

The advent of million-volt X-ray equipment brings X-ray radiography within the range of metal penetration and effective quality of radiation with radium. However, the experience of industrial applications has shown that the use of radium had expanded in spite of the availability of million-volt X-ray units.

Radium has some advantages in its use which will always be attractive to industry. A few advantages of radium over an equivalent X-ray unit are: low cost per unit examination for low volume manufacturers; availability without the first cost of expense of equivalent X-ray installations; and the flexibility of use with the small capsule of radium. It is probable that special applications would indicate further specific advantages in the use of radium over equivalent X-ray installations.

X-raying Spot Welds:

There are numerous applications of X-ray examinations which require special treatment, both as to the equipment used and technique developed. An important new field for X-ray examination has developed with the increased use of spot welding of thin aluminum sheet. The advantages obtained by welding aluminum aircraft materials is well recognized by designing and production engineers provided there is no sacrifice in strength and reliability in its use. In the development and control of aluminum welds the X-ray inspection method is extremely important. The areas involved in a spot weld are small and the defects that do occur require a degree of resolution on the film not common to most radiographic processes.

Figure 7 shows a poor aluminum spot weld. Cracks in spot welds are restricted according to certain specifications and can be controlled by proper regulation of the welding conditions. The structural variations occurring in an average spot weld are also indicated in Fig. 7. Other indications and types of defects will show radiographically if they affect the X-ray absorption. Lack of fusion in a spot weld can be determined radiographically by observing the disappearance of the characteristic dark ring which is the actual weld diameter or the diameter of the slug obtained on testing to failure.

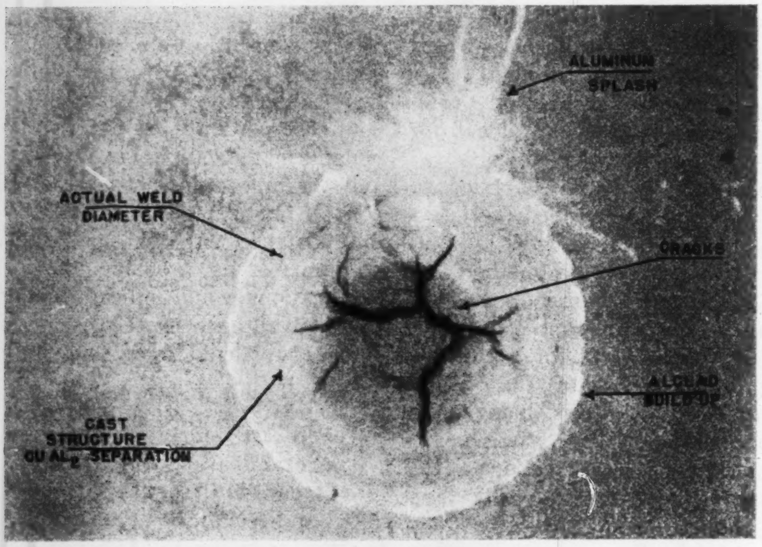

Fig. 7.—Aluminum Alloy Sheet Spot Weld Showing Radiographic Structural Detail.

Due to the thinness of most aluminum alloy spot welds, an X-ray beam of 15 to 30 kv. is required to obtain sufficient radiographic contrast on the films. Examination of the metallographic structure of aluminum alloy spot welds indicates that a high radiographic resolution would be desirable. However, the size of most of the defects occurring in the weld zone are such that visual observation of the X-ray films is sufficient. Examination of the X-ray films at higher magnifications for finer structural details will require consideration of the factors affecting radiographic sharpness.

X-ray technique for obtaining maximum detail and sharpness on the film requires the use of a low-voltage X-ray beam, small focal area X-ray tubes, and high-contrast, fine-grained films. The results obtained will depend upon individual technique and the care in observing the dark-room processing rules. Due to the increased exposure time required by the use of low-voltage X-ray beams and the slower fine-grained films, consideration should be given to the degree of resolution required for proper inspection.

The choice of the X-ray unit used for X-ray examination of spot welds will depend upon the relative low voltage X-ray output and the size of the focal target area required for the particular installation. A comparison of the effect of focal target area on resolution has indicated that, for visual examination only, the size of the focal area is unimportant. Examination of spot-weld films with a magnifying glass, or by projection, will require X-ray tubes with as small a focal target area as possible.

Microradiographic Technique:

A natural extension of the radiographic process appears in the attempts of various workers to resolve structural details in metals and other objects by X-ray absorption, formerly confined to the optical microscope. This process, called microradiography, has shown some advantages in application over pure optical reflection methods.[4] The technique required to prepare metal samples for microradiography is difficult until a convenient method has been worked out. It should not require any more time and trouble than the usual sample-polishing methods required for metallographic examination.

Microradiographic samples must be ground down to a thickness that is approximately of the order of magnitude of the structural differences existing in the metal sample in order to secure sufficient detail and sharpness in the film image. The procedure of sample preparation is generally decided by personal convenience and dexterity. A few suggestions are offered based upon the experience of one laboratory.

The sample is cut from the test object, by hand sawing or on the rubber wheel, as thin as possible to avoid subsequent labor. A section $\frac{1}{8}$ in. thick is considered a good starting point. The sample is then ground and polished on one surface by hand and the excess metal removed entirely from the opposite side by any convenient method. One method is to mount the smooth face of the sample on the flat ground face of a small steel block and remove the bulk of the metal by hand filing or by surface grinding. A suitable mounting material such as DeKhotinsky cement is required to hold the thin sample on the steel block against the grinding or filing action. An alternate method may be to mount the small flat sample in a transparent plastic material in a suitable molding press. After the sample has been filed down to approximately 0.010 in. thick it is removed from the steel block, described above, and ground down to the required thickness by hand on abrasive paper. As the sample is too thin to be held in the fingers for the sanding operation, some holder must be devised. A simple holder may be made by using a No. 9 rubber stopper. The sample may be made to adhere to the rubber stopper by coating the polished side with a thin layer of rubber cement which has been diluted with three times its volume of benzol or other thinner. Samples $\frac{1}{2}$ in. square have been ground flat to 0.001 in. thick with very little trouble by this method.

The application of the microradiographic technique depends to a large extent upon the proper understanding of the physical factors involved. Detail registered on the film or plate is entirely due to differences in absorption of the X-ray beam passing through the sample. Low-voltage radiation, in general, will give the greatest difference in absorption between constituents in the sample. However, more specific information can be obtained by taking advantage of the monochromatic X-radiation generated from different target materials in the X-ray tube and by the characteristic absorption edges of the different ele-

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[4] G. L. Clark and W. M. Shafer, "The Technique of Microradiography and Its Application to Metals," *Transactions*, Am. Soc. Metals, Vol. 29, September, 1941, p. 732.

FIG. 8.—Eutectic Type Oxide Inclusions Shown in a Steel Sample Microradiograph. No attempt to use the characteristic radiation for selective absorption (× 100).

FIG. 9.—Same Sample Shown in Fig. 8, Using the Selective Absorption of the Characteristic Radiation to Give Specific Information (× 100).

ments in the sample. Figures 8 and 9 show the microradiograph of an identical steel sample taken by utilizing different characteristic monochromatic wave lengths produced by different X-ray tube targets.

An eutectic oxide structure is shown in both Figs. 8 and 9. However, Fig. 9 shows a rather faint and diffuse white outline indicating a segregation by an atom having a characteristic absorption edge in the wave length region of the monochromatic X-ray beam. This absorption effect still persists in reduced amount by using a radiation only slightly shorter in wave length than that used in Fig. 9. Radiation in which the principal wave length is either longer or shorter than described above does not show this segregation.

Other uses for the microradiographic technique are only limited by the skill and initiative of the operator. Figure 10 shows the condition obtained when heavy elements and light elements are both present as a dispersed phase in a leaded steel sample. The white particles are free lead and the dark particles are sulphide inclusions. Microscopic examination of the sample was very confusing.

Fig. 10.—Microradiograph of Lead Steel (× 100).

Examination of the light metals by the microradiographic method is particularly easy and plates can be made showing considerable contrast and detail. Figure 11 is a section of an aircraft cylinder head adjacent to a weld area. An entirely different picture is obtained with the coarse cast structure of a 4 per cent copper, aluminum alloy shown in Fig. 12.

Considerable controversy exists at present as to whether the microradiographic method applied to the light

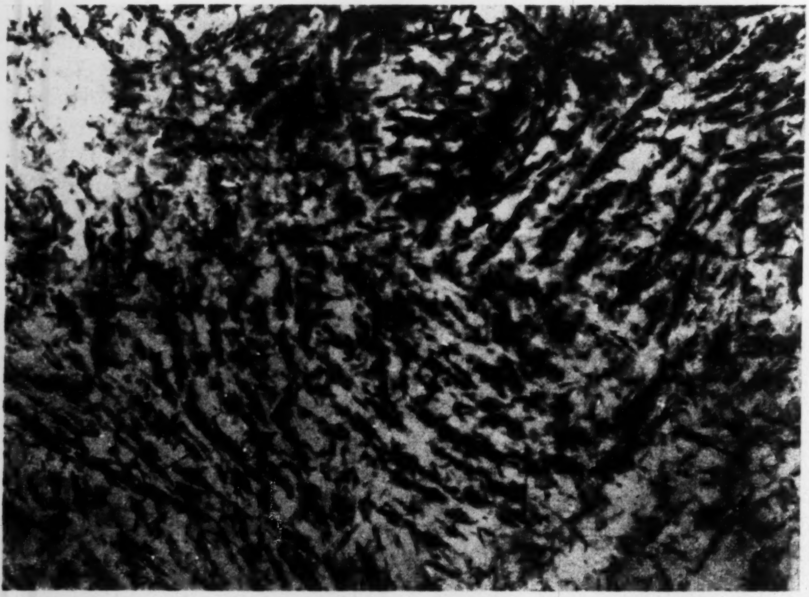

FIG. 11.—Microradiograph of Aluminum Cylinder Head Adjacent to a Weld Area (× 100).

The sample used was 0.015 in. thick giving more complete examination due to the added depth, than possible microscopically.

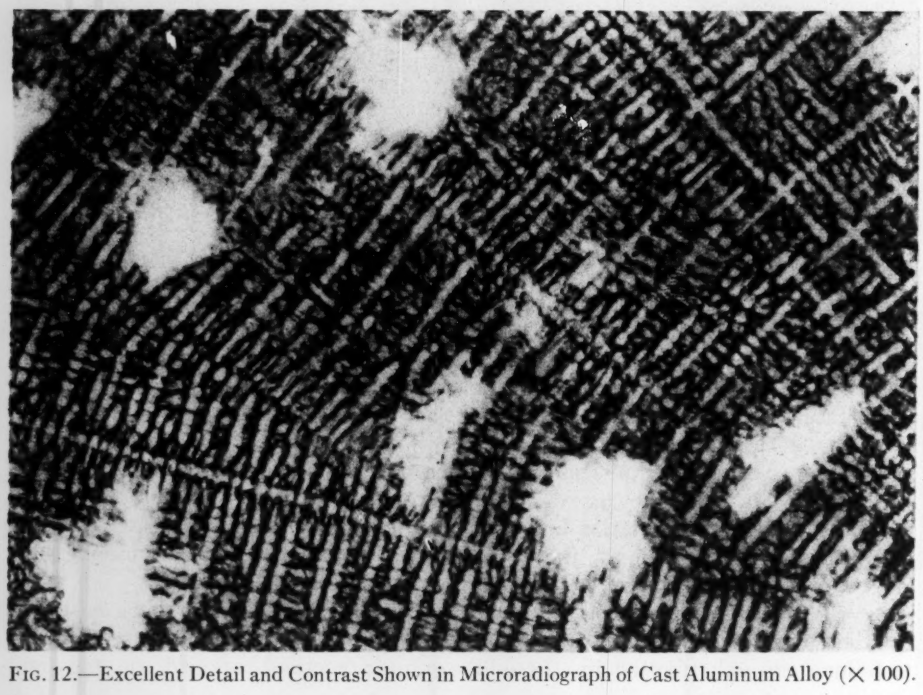

FIG. 12.—Excellent Detail and Contrast Shown in Microradiograph of Cast Aluminum Alloy (× 100).

alloys gives any information not obtainable by other methods. Specific applications utilizing this method of investigation will undoubtedly be used where theoretical consideration has been given to its full possibilities. For example, microradiographs of a series of aluminum alloys to which increasing amounts of an addition agent had been added gave a clearer picture of the structural changes occurring than the single cross-section used for microscopical study. Utilization of possible characteristic absorption effects have not been reported sufficiently in the literature to establish the possibilities of this method to light metal alloys.

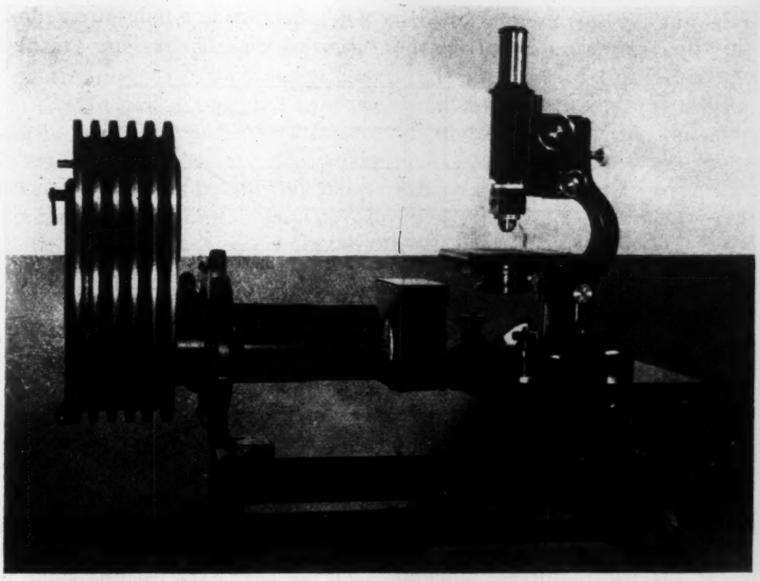

Fig. 13.—Projection Microscope Used for Viewing Microradiographic Plates.

Choice of films for registering the fine detail of the microradiographic process will depend upon the magnification one wishes to use to examine the resulting image. For magnifications of 50× or over, the Eastman Kodak Co. high-resolution emulsion on plates or films is recommended. Development of the high-resolution plate is most conveniently done in standard X-ray developer for 2 to 4 min. An alternate method of developing is to use Eastman X-Ray Replenisher solution, adding sufficient potassium bromide to restrain chemical fogging. Use of the undiluted replenisher solution for development of high-resolution plates allows a reduction of the exposure time up to a approximately half the time required for the standard X-ray developing solution. Fresh solutions must be used for each plate to prevent staining and irregular results.

Considerable improvement in results may be obtained by proper viewing of the microradiographic images at the higher magnifications. Figure 13 shows a microscopic unit ideally designed for viewing microradiographic plates. Experience has shown that best results will be obtained with an image density between three and five. This requires a strong viewing light which is obtained by the tungsten arc lamp used on the unit illustrated. A substage condenser of the Abbé type is used to focus the light onto the plate being examined under the microscope. A condensed light beam for viewing purposes increases both the clarity and apparent contrast of the images under the microscope.

SENSITIVITY OF THE X-RAY PROCESS

Methods have been previously discussed in this paper to control the amount of scattered radiation reaching the film by the use of filters, blocking methods, and X-ray beam diaphragms. However, the detection of small defects is still limited by the image sharpness resulting from radiation other than from a point source. The eye can detect small differences in density provided the difference in density is rather sharp.[5] Broad focal spot X-ray tubes diffuse the image of small defects which readily escape detection by eye even with an appreciable difference in density.

The method for observing the sensitivity of the X-ray process as universally adopted consists in placing a penetrameter gage on top of the section being examined. The dimensions and design of penetrameter gages is specified by national technical societies and by Government specifications. In general, the gages used consist of material of the same composition or absorption as the part being X-rayed with a thickness of 2.0 per cent of the metal being examined. One or more fine holes are drilled in the gage which must register on the film for acceptable radiographic technique. The 2.0 per cent gage thickness can be easily registered on the X-ray film with average X-ray technique. The small holes in the penetrameter are often difficult to distinguish on the X-ray film unless particular attention is paid to image sharpness and maximum film contrast. Actual sensitivity of the radiographic process is in the ratio of the smallest thickness or dimensions of a defect which can be observed on the film to the thickness of adjacent sound metal. The determination of actual sensitivity can be approximated in any section by the use of artificial defects such as drilled

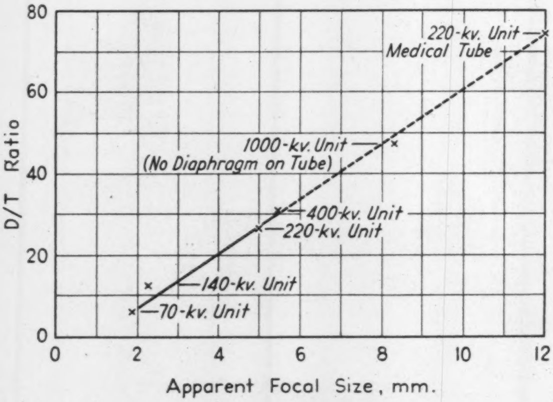

FIG. 14.—D/T Ratio Experimentally Determined for a Number of X-ray Units.

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[5] R. Glocker, "Materialprüfung mit Röntgenstrahlen," p. 71, Julius Springer, Berlin (1936).

holes or slots or by the use of a metal wedge containing milled grooves of various depths. Penetrameter or gage sensitivity should only be considered as a check on the technical factors of the radiographic process.

The sharpness of radiographic detail is determined by the size of the X-ray tube focal area and the relative distance between the tube and object and X-ray film and top of the object. A number of methods may be used to determine the optimum distance ratios or *D/T* ratio, where *D* is the tube to object distance and *T* is the film to top of object distance.[6] A standard artificially built-up test block has been devised by Lester for determining this ratio. The ratio can best be determined by the purchaser of individual X-ray units. Figure 14 shows the *D/T* ratio experimentally determined for a number of X-ray units.

As an example, a steel casting is to be X-rayed on a 220-kv. unit with the film to top of object distance equal to 3.0 in. The curve in Fig. 14 shows that a tube distance of 78 in. is required to produce acceptable image sharpness on the film. General practice in X-ray inspection laboratories in the past has been to use an object to tube distance which would give an exposure time convenient to operating conditions without too much regard for image sharpness. The curve in Fig. 14 shows that a Medical Therapy X-ray tube, with focal area of 12 by 12 mm., would require prohibitively long tube to work distances to secure adequate image sharpness.

The focal target area of the million-volt X-ray unit can be varied to suit the electron energy input required for the exposure. For low milliamperage requirements, a focal area as small as 2 mm. in diameter may be used. Actual measurement of the focal area is difficult and requires rather complicated equipment. The *D/T* ratio given for the million-volt unit in Fig. 14 was experimentally determined by a test object as described above. The point on the curve of Fig. 14 is for the maximum milliamperage used with the million-volt unit and hence the largest focal area. Actual tests with two different focal spot measuring devices agree very well with the experimentally determined equivalent focal area.

The radiographic sensitivity obtainable in practice can be roughly determined by a theoretical study of those factors which affect the film-viewing conditions; the variables contained in the simple absorption equation

$$I/I_o = e^{-ut}$$

where:

I_o = primary X-ray beam intensity,
I = transmitted X-ray intensity,
u = absorption coefficient,
t = section thickness, and
e = base of natural logarithm;

and the effect of scattered radiation in the object or adjacent surfaces. A more practical approach to the problem of securing maximum sensitivity in the radiographic examination of complicated sections for the average commercial laboratory may be by the trial-and-error method. An understanding of those physical conditions that produce undesirable results in a radiograph is of invaluable aid in establishing technique by the trial-and-error method. Some of these undesirable factors will be considered in the following papers of this Symposium.

One of the most useful aids in determining the quality of a radiograph of a complex casting is the tungsten wire grid. The grid is constructed by winding tungsten wire 0.010 in. in diameter around a thin flat frame of suitable dimensions in a close spiral. Tungsten wire is recommended as it is quite opaque to the wide range of voltages required in general radiographic work. Placing the tungsten grid on top of the casting during the test exposure will give indications on the film of most of the undesirable effects ordinarily encountered in radiography. An example of the use of the tungsten grid on a light alloy casting is shown in Figs. 3 and 4. The scattering effect is markedly reduced in X-raying this casting with a 0.5-mm. copper filter at the X-ray tube port. The original X-ray film shows that an actual increase in defect detail has been obtained by the use of proper filtration of the primary beam. Lack of primary beam penetration as shown by the disappearance of the grid wire image on the film is very useful in determining the completeness of the radiographic exposure. A common mistake in radiographic inspection is the failure of the X-ray operator to determine the true limits of primary beam penetration in complicated shaped castings.

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[6] H. H. Lester, "Some Aspects of Radiographic Sensitivity in Testing with X-rays," ASTM Bulletin, No. 100, October, 1939, p. 33.

Considerable disagreement exists in the determination of radiographic sensitivity. Use of the penetrameter gage serves a useful purpose but the results must always be expressed in terms of gage sensitivity. A large amount of information on sensitivity of the radiographic process, both from a theoretical and practical standpoint, has been accumulated in recent years and it is expected that a reliable and universally adopted procedure will result.

CONCLUSION

The radiographic process as applied to war production problems has been reviewed. Application of X-ray inspection has multiplied many times in the past year, and will undoubtedly increase as the special demands of the armament program increase. Some practical applications to large-volume production inspection have been discussed for both low-voltage and high-voltage X-ray practice. Special applications of X-ray examination as in spot welding and microradiography have been given considerable attention, and new methods and applications developed for the latter purpose are outlined.

The experience gained on present X-ray examination of high-strength castings indicates a possible increased use of X-ray inspection on high-strength castings for future peace-time purposes. X-ray equipment which may be available in the future will include small-size and multi-million-voltage outfits. The known methods of accelerating electrons to high velocities without high potentials give a hint as to possible future developments.

DISCUSSION

Mr. Robert Katz.[1]—You mentioned that steel castings are inspected by X-ray and magnetic methods. I wish you would expand on that, because of some past controversy in this field.

Mr. Don M. McCutcheon.[2]—Cast steel parts for use in aircraft are designed and heat treated to produce the best strength for weight-saving purposes. They must be cast as sound as possible, and must also be free from any defect, such as cracks which might be inherent in the hardening or strengthening methods. The X-ray method is useful in determining and checking on the casting methods, and the magnetic inspection method is essential for those types of defects common to the process of hardening or strengthening steel. They are not to be confused as overlapping or detecting the same type of defects. The use of X-rays should be limited to its special field.

Mr. Otto Zmeskal.[3]—What developments can be expected in the line of registering devices? Can we expect the increased use of the Geiger counter? Will we have higher and higher sensitivity films, or are there limits in those directions?

Mr. McCutcheon.—The developments of the future may surprise us. The X-ray method as used at present is almost entirely photographic. I know of a number of attempts to register defects electrically, but they have not been too successful.

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[1] Army Air Forces, Wright Field, Dayton, Ohio.

[2] Metallurgical Engineer, Main Laboratory, Rouge Plant, Ford Motor Co., Dearborn, Mich.

[3] Assistant Professor of Metallurgy, Illinois Institute of Technology, Chicago, Ill.

RADIOGRAPHY OF WELDS AND WELDMENTS

By R. E. Lorentz, Jr.[1]

Synopsis

The material in this paper consists of developments in the general art of radiography of welds which have come into use since 1936 when an earlier Symposium on Radiography and X-ray Diffraction Methods was held by this Society. The excellent papers given at that time represented the best practices in use. Since that time, a number of developments have been made which are covered in the present paper.

The maximum available voltage has been raised from 400,000 v. to 1,000,000 v. This development has allowed many further improvements in technique. Not only has it raised by many inches the maximum weld thickness which can be radiographed, but it also has allowed the wider use of lead intensifying screens to improve detail in the exograph. It also gives superior techniques for many special applications.

New fine-grained, high-contrast films have come into use which improve greatly the detail of exographs and gammagraphs. Techniques such as double-film viewing, which present many advantages, have come into use, and the trend toward longer film lengths for special cases is discussed.

There have been some changes in code requirements which have come about as a result of wider experience, and these changes are discussed.

In 1930, the United States Navy in accepting fusion-welded boiler drums made radiographing mandatory for nondestructive testing of the main welds. In 1931, the A.S.M.E. Boiler Code Committee accepted fusion-welded pressure vessels, making radiographic inspection of the main welds mandatory for vessels of hazardous service requirements. Since that time, a large number of other codes have written X-ray inspection into their specifications, wherever possible loss of life or serious property damage might result from a failure due to faulty welding. The experience gained through many years has confirmed the necessity for this type of nondestructive testing. Notwithstanding the tremendous strides made in welding, there still exist a large number of variables hard to control, not only in manual welding, due to the human element, but also in automatic welding. Not infrequently, severe defects are detected and repaired. The excellent service records of vessels so inspected has done much to further the use of welding and instill confidence in its results.

Qualification tests of welding operators show that these operators can make a good weld under fairly ideal conditions. It does not assure, however, that day in and day out, regardless of the operator's physical condition, he can or will consistently obtain such quality. Similarly, when a welding procedure is qualified, it is no guarantee that that procedure will give consistently high-quality results, for some variables, of

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[1] Metallurgist, Combustion Engineering Co., Inc., Chattanooga, Tenn.

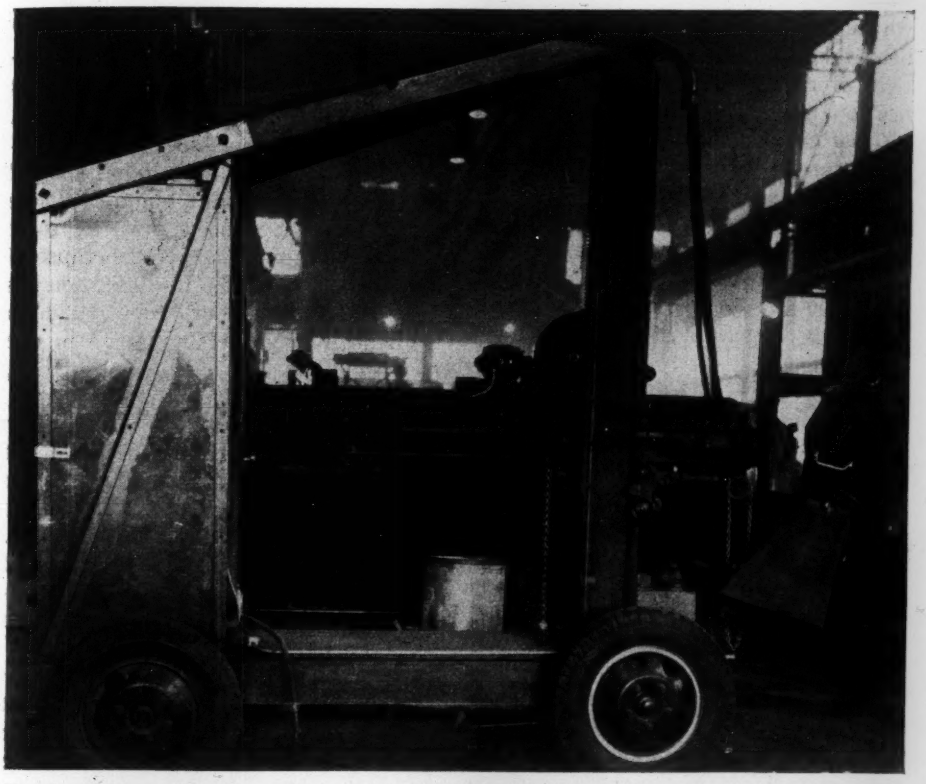


FIG. 1.—220-kv.p. Machine. First produced in 1938. Note portability and universal adjustments

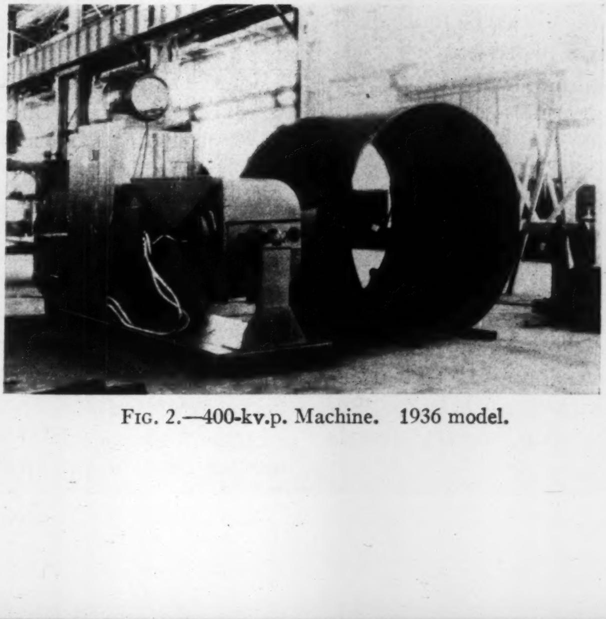


FIG. 2.—400-kv.p. Machine. 1936 model.

which there are many, may not have been taken into account in drawing up that procedure. The procedure qualification is definitely helpful, but is no 100 per cent guarantee of weld quality. Consequently, where welds must withstand severe service requirements, as they are doing today in pressure vessels and various ordnance parts, the X-ray inspection is very justifiable. This is especially true for welds on air-hardening steels, of which there has been an increasing use.

Today, the Navy, the Army, the Coast Guard, the American Bureau of Shipping, the American Society of Mechanical Engineers, various insurance agencies, and chemical concerns, among many others, have formulated specific X-ray requirements. Many times, controversy arises over various borderline defects, and it is in times such as these that controversy results in more definite specifications as to allowable defects. Obviously, the ideal X-ray inspection is one that does not vary from one inspector to another. The fulfillment of this, in turn, rests upon the closer classification of borderline defects, the improvement of X-ray techniques so as to obtain the clearest or most easily interpreted radiographs, and many other factors, all of which are greatly dependent upon experience. It is questions such as these—not only in X-ray specifications, but in all specifications—that are being ironed out in these times.

At the 1936 Symposium on Radiography and X-Ray Diffraction Methods, a number of very excellent papers on radiography were presented. A few developments since that time are presented in this paper. There has been a fairly voluminous literature on various phases of the subject since 1936. The principles, in the main, however, remain the same.

APPARATUS

One of the largest single developments was the recent development, by General Electric Co., of a portable 1,000,000-v. industrial X-ray machine. This development, a large one in itself, has allowed further refinements in techniques. These refinements have been known for some time, but not generally used because of voltage limitations. This limitation has now been eliminated, the maximum available voltage being jumped in one step from 400,000 v. to 1,000,000 v., with consequent far-reaching effects.

The industry today is fortunate in having such a wide range of equipment available, ranging from 5000 v. for microradiographic work to 1,000,000 v. for radiographing welds in heavy plate. In addition to this increase in range of available voltages, the over-all portability and adjustability of the machines has been somewhat improved.

Figure 1 shows a 220-kv.p. machine in use for radiographing relatively light thicknesses. This particular machine was first produced early in 1938. The position of the tube can be universally adjusted and the entire machine can readily be moved to almost anywhere in the shop. A 250-kv.p. machine of the same general design has been in use since 1940.

Figure 2 shows a 400-kv.p. machine in use since 1936. This machine can be moved fixed distances along its tracks and has sufficient positioning adjustments for most purposes. A later model of this machine has been developed in which the tube is self-rectifying. A better wave form exists, allowing slightly better radiographs. This machine is shown in Fig. 3. These machines are all oil insulated and of shockproof design.

Figure 4 shows a 1000-kv.p. machine produced in 1941. This machine is

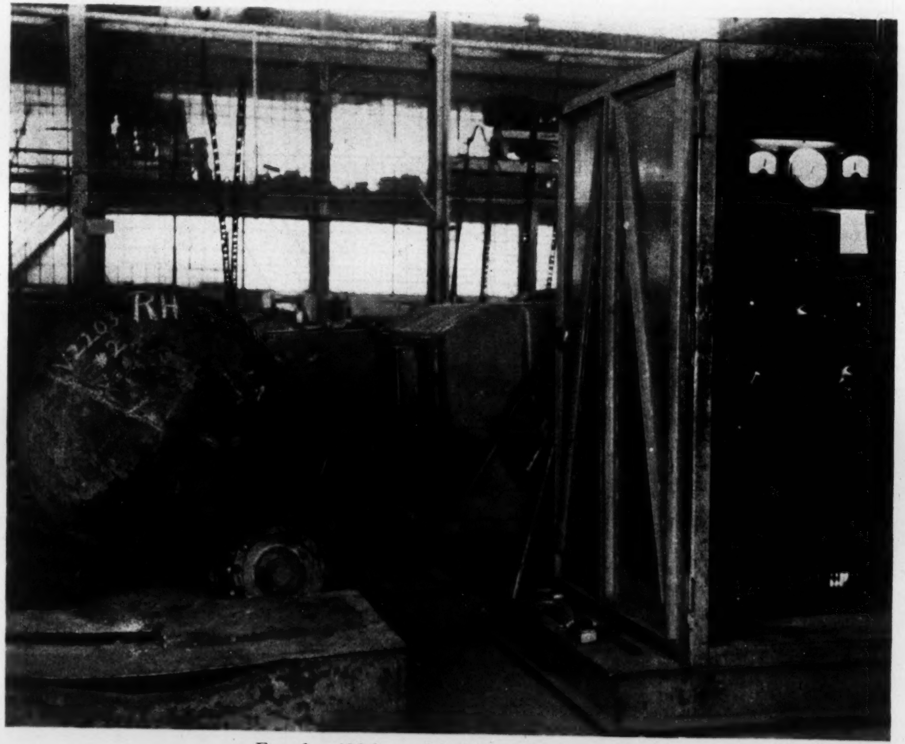


FIG. 3.—400-kv.p. Machine. Later model.

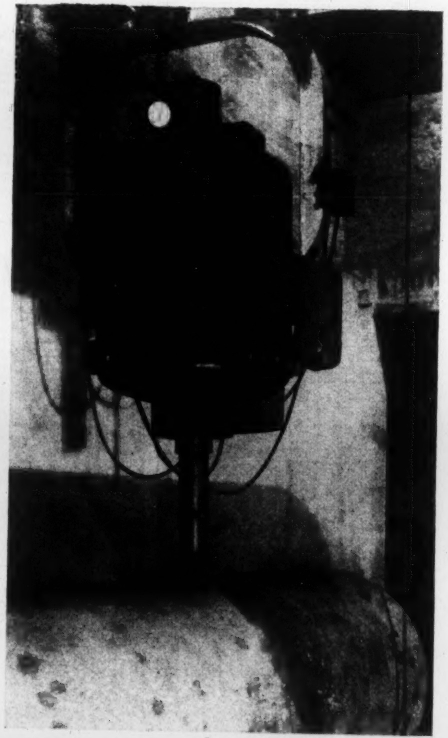


FIG. 4.—1000-kv.p. Machine with Especially Good Adjustability.

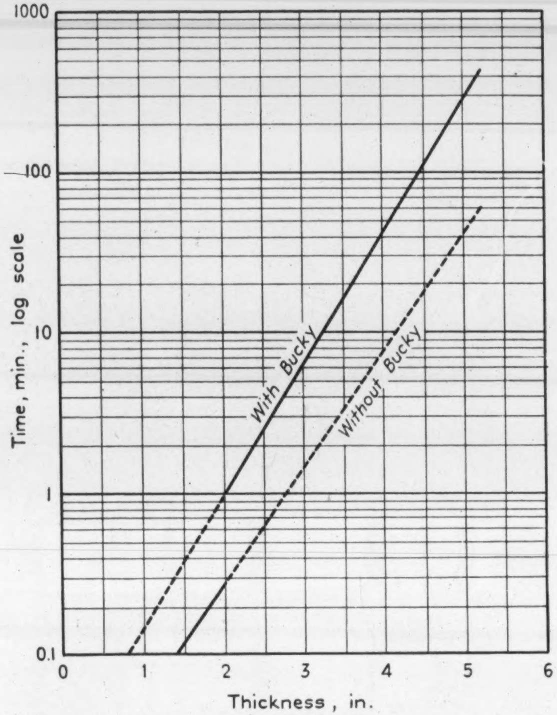


FIG. 5.—Exposure Time Required by 400-kv.p. Machine With and Without Bucky Diaphragm.

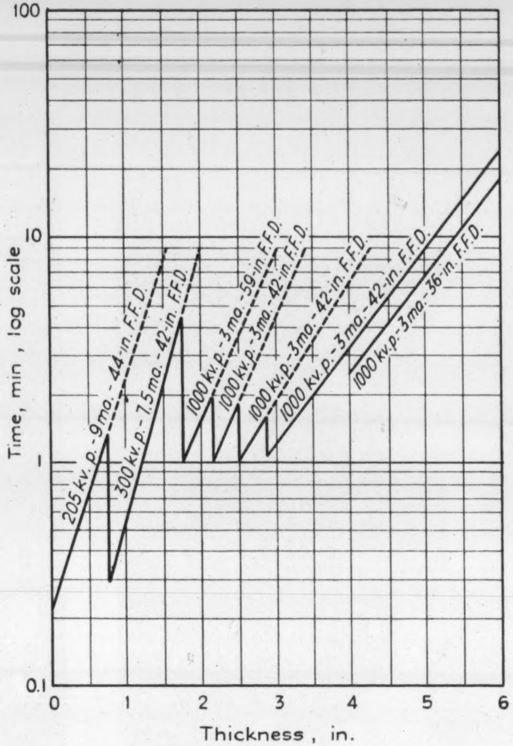


FIG. 6.—Composite Technique Chart.

Lead intensifying screens, high contrast fine-grained film, $\frac{1}{4}$-in. lead filtration, $1\frac{3}{4}$ to $2\frac{7}{8}$-in, $\frac{1}{8}$-in. lead filtration above $2\frac{7}{8}$ in , double film viewing above $2\frac{1}{2}$ in.

housed in a special concrete building. It has especially good positioning adjustments. The entire unit is assembled in a cradle carried by an overhead crane. It can be traversed in three directions by the crane, can be rotated 360 deg. on the yoke of the cradle, and can be rotated on trunnions in the cradle from the vertical to the horizontal position.

With this range of equipment, welds in steel up to 6 and 7 in. in thickness can be radiographed in a reasonable time.

TECHNIQUE

In the recent past, when 400,000 v. was the highest available for industrial work, it had become the practice in our shops to use a Bucky diaphragm at thicknesses of 2 in. and above, in the interest of clarity of exograph. This diaphragm eliminated much of the scattered radiation, but, at the same time, considerably increased exposure times. Suitable exographs on heavy plate, however, could not be obtained without it. Figure 5 shows a semi-log graph showing exposure times required by a 400-kv.p. machine with and without the Bucky diaphragm. It can be seen that at about 5 in. of thickness, exposure times become excessive. Only last year, it required $7\frac{1}{2}$ hr. to X-ray 12 in. of weld in a $5\frac{1}{4}$-in. plate. With the advent of the 1000-kv.p. machine, these exographs were obtained in 10 min. and the use of the Bucky diaphragm was virtually eliminated, all thicknesses at which it would ordinarily be used being radiographed on a 1000-kv.p. machine. At the same time, it is now possible to radiograph much heavier weld thicknesses, and this in a reasonable time.

It has been general practice to use calcium tungstate, or fluorescent, screens in combination with industrial X-ray films in order to keep exposure times at a minimum. Especially at the lower kilovoltages, these screens have a high intensification factor. It had been recognized that lead screens, in combination with no-screen (direct-exposure) film, would give better quality exographs, but because of their lower intensification factor, did not find general use at voltages up to 400,000 v. With the advent of the 1000-kv.p. machine, however, the lower kilovoltage machines could be used for radiographing considerably lighter thicknesses for which they had more than ample power. The road was then paved for use of lead screens, their lower intensification factor being offset by use of higher voltages. The increased clarity of detail is considerable. At one manufacturer's plant using this technique, welds up to $\frac{3}{4}$ in. are radiographed with 220 or 250-kv.p. machines; welds from $\frac{3}{4}$ to $1\frac{3}{4}$ in. are radiographed with 400-kv.p. machines; and welds above $1\frac{3}{4}$ in. are radiographed on a 1000-kv.p. machine. Front screens 0.005 in. thick and back screens 0.010 in. thick are used in combination with fine-grained, high-contrast film, recently marketed by film manufacturers. Very excellent results are obtained with this combination on all machines, at exposure times for welds up to $1\frac{3}{4}$ in. thick but slightly longer than those used before with tungstate screens. For welds greater than $1\frac{3}{4}$ in. thick, the exposure times required are much less than before. Figure 6 shows a composite technique chart for thicknesses from 0 to 6 in. It can be noted that a filter between the plate and the film is used on the 1000-kv.p. machine. This filter is necessary at the heavier thicknesses to stop the scattered and secondary radiation from the plate.

Figures 7 to 11 show a series of exographs containing typical defects in material $\frac{1}{4}$ to 4 in. thick. These exographs were taken using the techniques described above.

The builders of pressure vessels, in the main, are fortunate in that the ma-

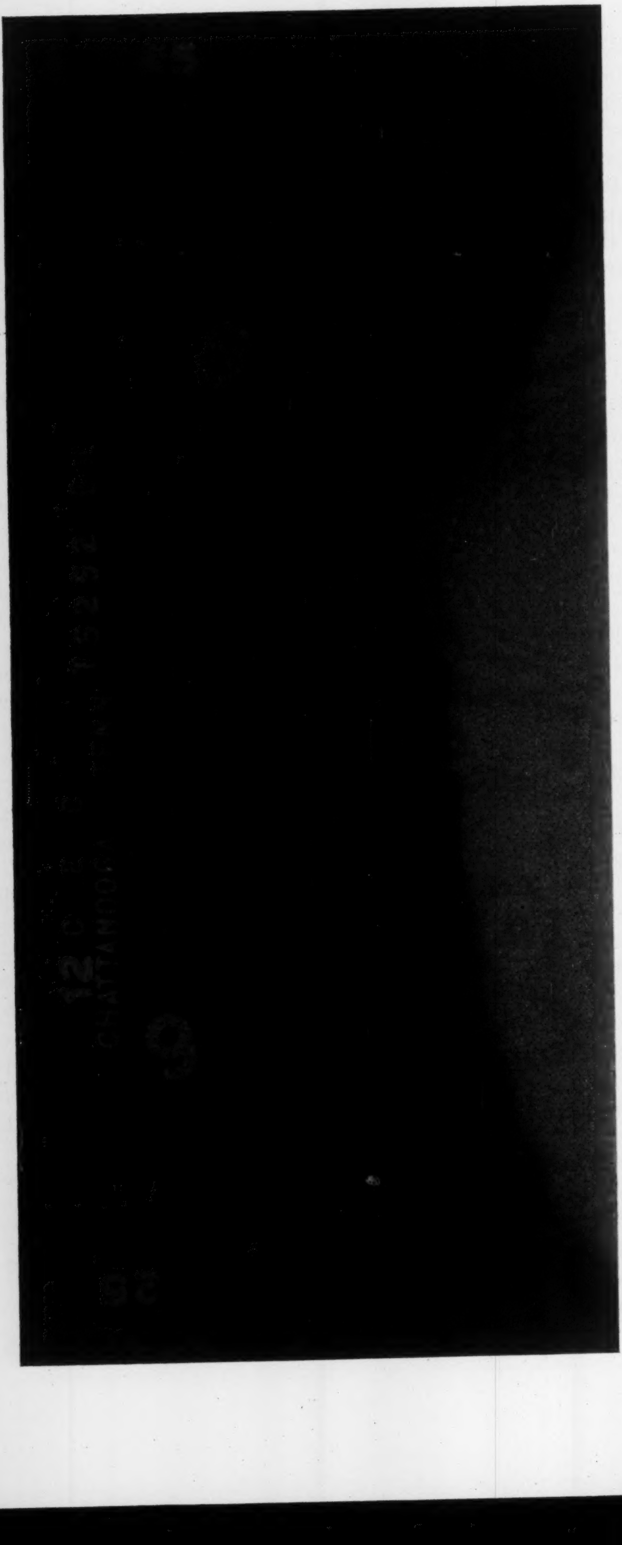


FIG. 7.—Cracks in Weld of Inserted Type Nozzle. Weld ¼ in. thick.

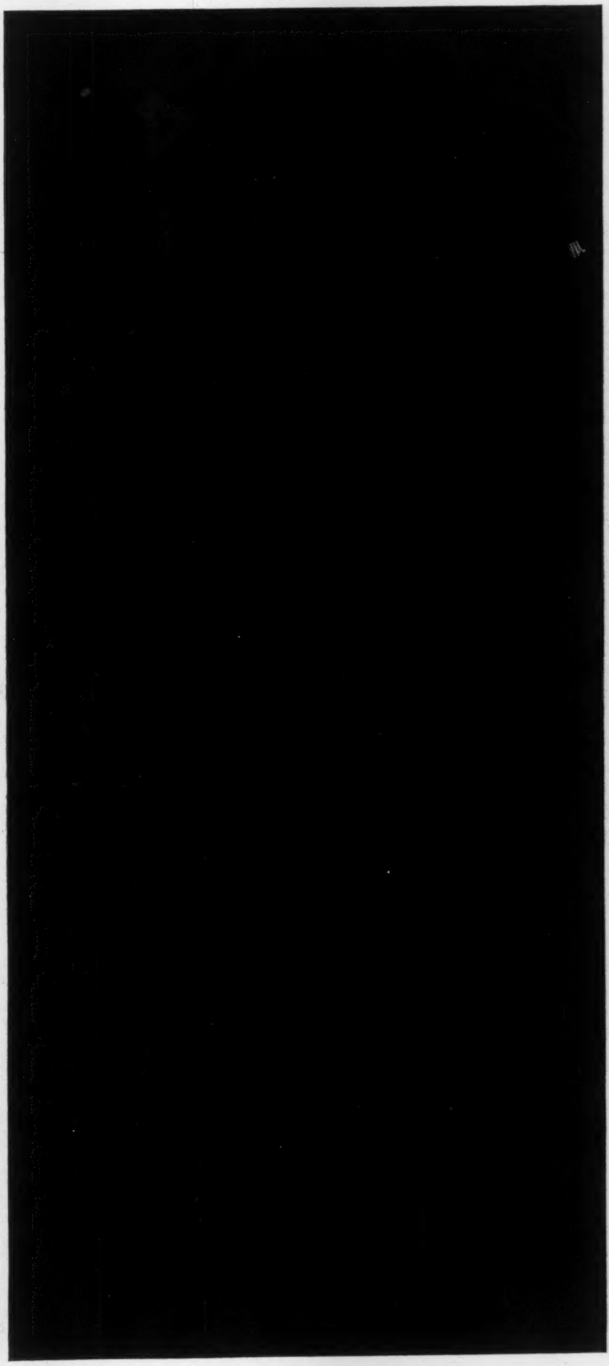


FIG. 8.—Cracks (Upper Left Middle) and Porosity Spots in Girth Seam Weld. Weld $\frac{7}{8}$ in. thick.

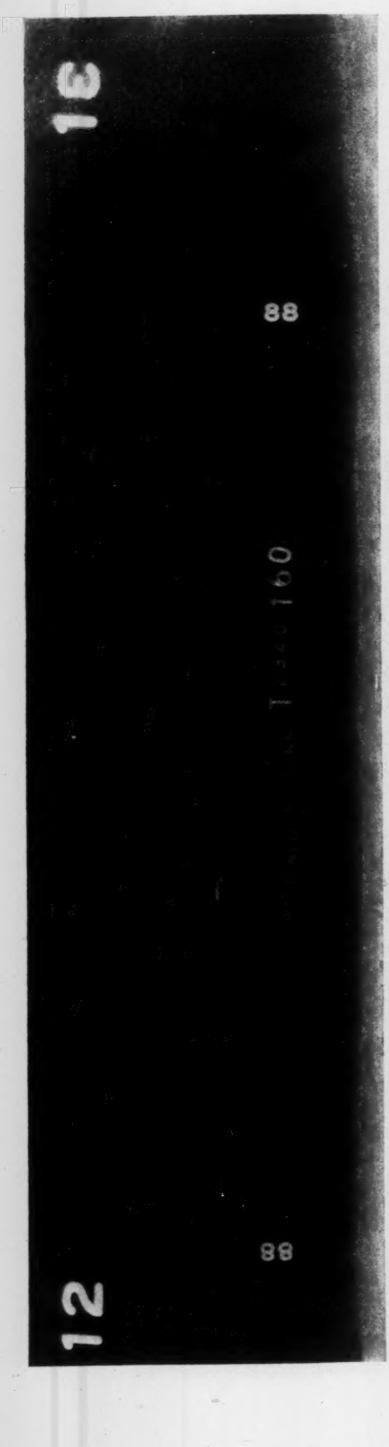

12
16
88
88
160

FIG. 9.—Long Crack (in Center) and Porosity Spots in Long Seam Weld. Weld $\frac{7}{8}$ in. thick.

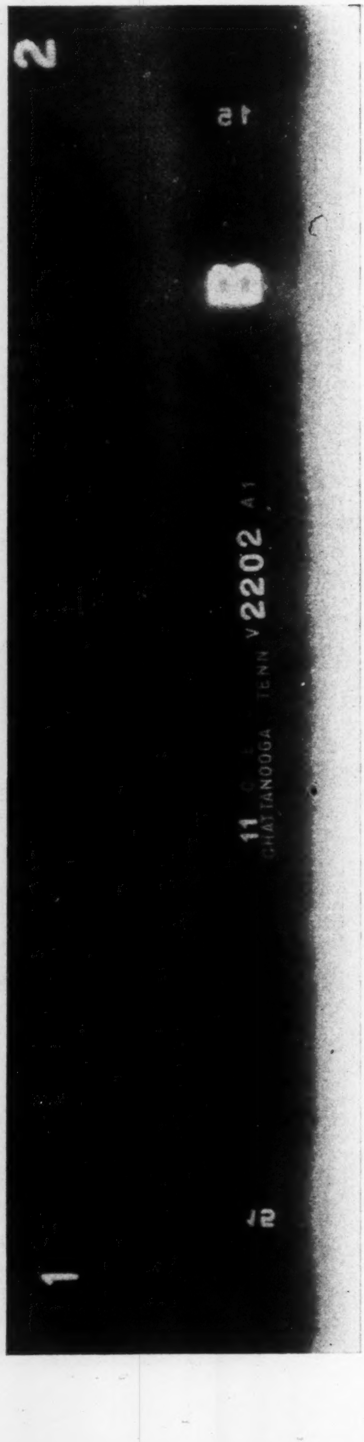

1
2
B
11
CHATTANOOGA TENN
2202
A1

FIG. 10.—Slag Inclusions (Extreme Left) in Long Seam Weld. Weld $1\frac{1}{2}$ in. thick.

FIG. 11.—Experimental Film Showing Test Block ⅛ in. Thick with Hairline Crack Placed on Top of 4-in. Plate. (Total thickness 4⅛ in.)

jority of welds are butt welds. These welds are usually ground flush with the plate and any flaws that are present are generally orientated such that a single direction X-ray is all that is required. Certain types of nozzle welds are sometimes required to be X-rayed. These welds may be of varying thicknesses, and in cases such as these, each job must be considered separately and a suitable technique worked out. The tube may be placed such that the rays pass through the thinner section at a greater angle; masking technique may be used to build up to uniform thicknesses with a material of suitable density; or a filter may be placed in the X-ray beam to decrease contrast. The various techniques are well known and have been well covered in the literature.

In order to insure that a film showing proper detail is being obtained, most codes specify a penetrameter or sensitivity gage which must show on every exograph. The A.S.M.E. Code penetrameter has become more or less standard. It consists of a piece of material approximately the same as that being X-rayed, which has a thickness of 2 per cent of the thickness of the weld being radiographed, and in it are drilled three holes of diameters 4 per cent, 6 per cent, and 8 per cent of the thickness of the weld being radiographed. These penetrameters are placed on the tube side of the weld, two for every film, and are placed such that one shows on each end of the film, where the angularity of the rays is greatest. The outline of the penetrameter and all three holes must show clearly to indicate a satisfactory technique. These penetrameters can be seen on the exographs of Figs. 7 through 11. Figure 12 shows the standard dimensions.

Previous to the use of this type of penetrameter, the A.S.M.E. Code specified the use of a step-type penetrameter. It consisted of a series of steps, each step representing 2 per cent of a certain thickness. One penetrameter was used up to 2-in. thickness, and another between 2 and 4 in. In use, the step representing 2 per cent of the thickness being radiographed should show up clearly. Actually, however, the steps representing heavier thicknesses than that being radiographed showed easily, and with these as a reference mark, it could be easily imagined that the proper step also showed with enough clarity. The new type penetrameter, being of a single thickness, does not have this disadvantage.

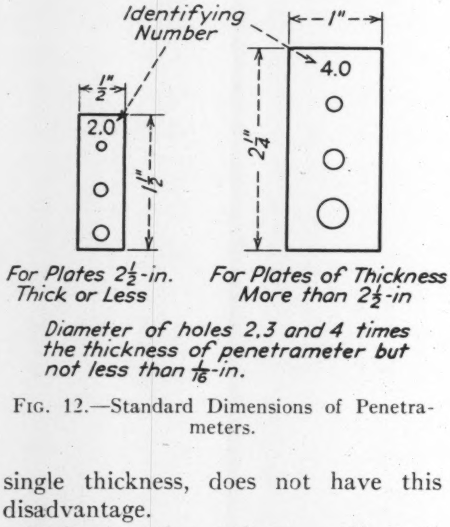

Identifying Number
1"
4.0
1/2"
2.0
2 1/4"
1 1/2"
For Plates 2½-in. Thick or Less
For Plates of Thickness More than 2½-in
Diameter of holes 2,3 and 4 times the thickness of penetrameter but not less than 1/16-in.

FIG. 12.—Standard Dimensions of Penetrameters.

It is general practice to radiograph pressure vessels before the stress-relieving operation. If a vessel, after welding, is out of round beyond the tolerance limit set by the codes, it is usually rounded up, radiographed, and then stress-relieved. With some alloy steels, however, the heat treatment temperature is high and the vessels sometimes sag in the furnace. This necessitates rounding up after the heat treatment, and the vessel is radiographed again after such rounding up to insure that no cracks were formed in the rounding

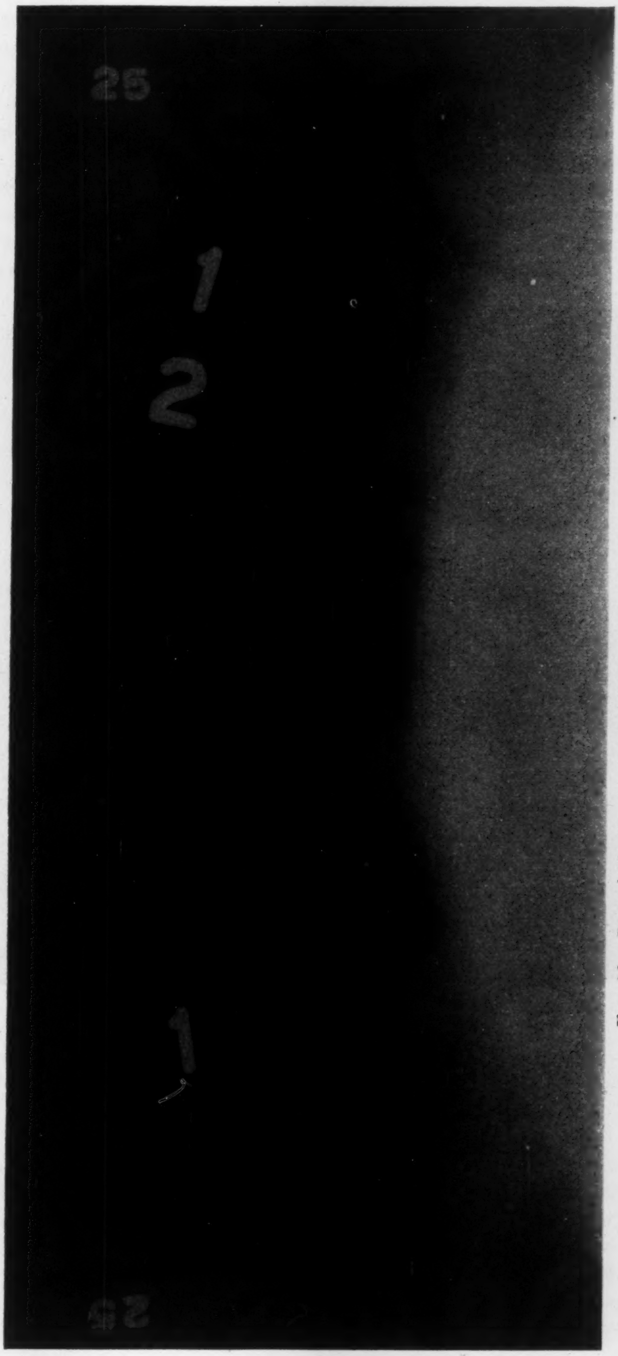


FIG. 13.—Round-Up Cracks Around Inserted Type Nozzle. Weld ¼ in. thick.

operation. Figure 13 shows an exograph with this type of crack.

It is general practice to use films 17 in. long and 3½ or 4½ in. wide, with the 4½-in. film showing preference in order to minimize retakes because of faulty centering. On girth or circumferential weld seams, films 10 in. long are generally used so that the film can be kept closer to the seam. These practices may or may not hold when radiographing with the new 1000-kv.p. machine. On the thinner thicknesses radiographed with this machine, the focal distance may be increased sufficiently so that film lengths of 34 in. can be used. The use of this technique depends upon whether the X-ray room is large enough to allow sufficient head room for the machine, so that a long enough focal distance to minimize angularity at the ends of the film can be obtained. The 1000-kv.p. machine is also used in the radiographing of girth seams with a single exposure. The focal spot of the 1000-kv.p. X-ray tube is at the outer end of a long cylinder extending from the tube head. The main beam of X-rays generated at the anode passes straight through it and other rays leave it in all directions. The focal spot can then be inserted into the manholes on either end of a drum, far enough so that the girth seam, holding the head to the main shell, can be radiographed from the inside out. With this technique, and wrapping film around the outside circumference of the welded seam, the entire seam can be radiographed in one exposure. The full advantages of this technique have not yet been fully realized because of set-up difficulties. Lead screens, without suit-

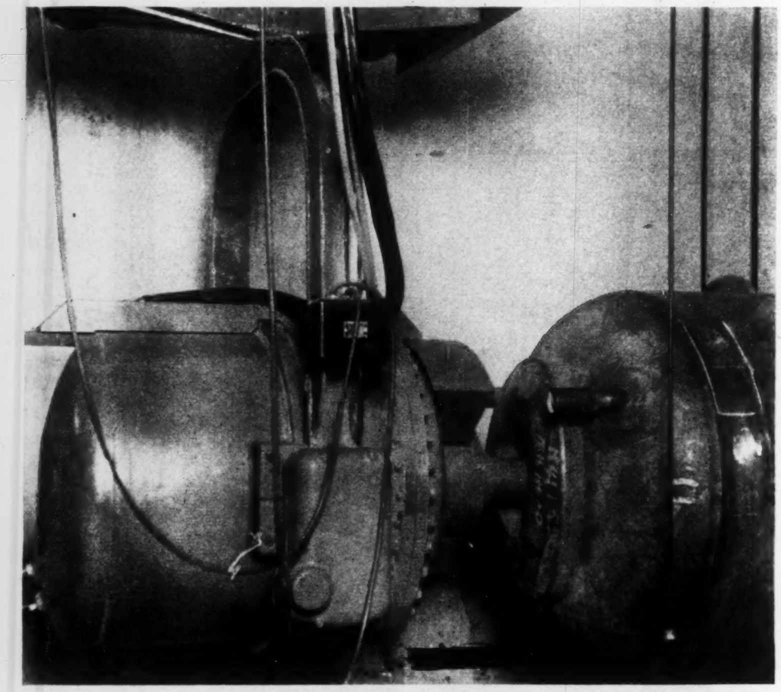


FIG. 14.—1000-kv.p. Machine in Position for Girth Seam Shot.

able backing, cannot be used because the bending wrinkles them and causes the film to have a mottled appearance. A lead-impregnated rubber sheeting, known as XRP sheeting, marketed by General Electric X-Ray Corp. has been tried with good results. Other difficulties, however, are that a filter of lead, copper, or brass, or some other suitable material, must be placed over the film and between the film and the weld seam to stop scattered radiation. The entire combination then requires a bulky set-up. No doubt, however, mechanical means to simplify this will soon be perfected. Figure 14 shows the machine in position to radiograph a seam such as this. Figure 15 shows two exographs of the same spot on $5\frac{1}{4}$-in. plate, one using the conventional lead screens and the other the XRP sheeting. Equal detail is present in both, but the exposure time required with the sheeting was twice that required with the lead screens. The 1000-kv.p. machine has a host of other advantages in special applications, but these will be covered in other papers.

There is another technique finding wide use, especially on the heavier thicknesses, a technique known as double-film viewing. An exposure is made with two films in the same cassette. Any defects present in the weld show on each film, and by viewing the films simultaneously, the defects can be picked out very nicely by sliding the films over each other until corresponding parts of the films are superimposed. This technique has many advantages. It eliminates retakes due to film defects, since any defects not present on both films are necessarily not weld defects. It has an advantage in radiographing differential thicknesses, since the darkest portion of the film can be interpreted by single-film viewing and the lightest by double-film viewing. It decreases exposure times, since the over-all required density is the sum of the densities of each film.

WELD DEFECTS

The subject of weld defects—which should be allowed and which should be removed—is still a controversial one. Obviously, a crack of any size should be removed, and the codes so specify. On the other hand, the questions of just exactly how much porosity is definitely detrimental or what slag inclusions can be allowed have not yet been answered to everyone's satisfaction. The A.S.M.E. Boilder Code at the present time judges acceptable or unacceptable porosity by comparison with a set of standard films. The borderline cases are shown in Fig. 16. This standard set does not take into account any variable of thickness. It is reasonable to conceive that more porisity than shown on the film may be tolerated in heavy welds than in thin welds. In the past, porosity in heavy welds was not as easily picked up in the exograph as it was in the lighter welds. This is the only basis on which the single standard of porosity is justified. In radiographing with 1,000,000 v., however, porosity is picked up very clearly in heavy welds, and this may justify a reconsideration of the code requirements.

Conversely, the present code allows a slag inclusion of length $\frac{1}{3}T$ where T is the weld thickness. These inclusions must be separated by $6L$ where L is the length of the longest inclusion, and their total length when added together must be not more than T in a length of $12T$. These requirements, then, would allow a slag inclusion $1\frac{2}{3}$ in. long in 5-in. plate. This is more likely to be dangerous than considerable porosity in this thickness. Similarly, if two slag inclusions of lengths $\frac{1}{2}$ in. each were present in a weld of this thickness and separated by $\frac{1}{2}$ in. of clean

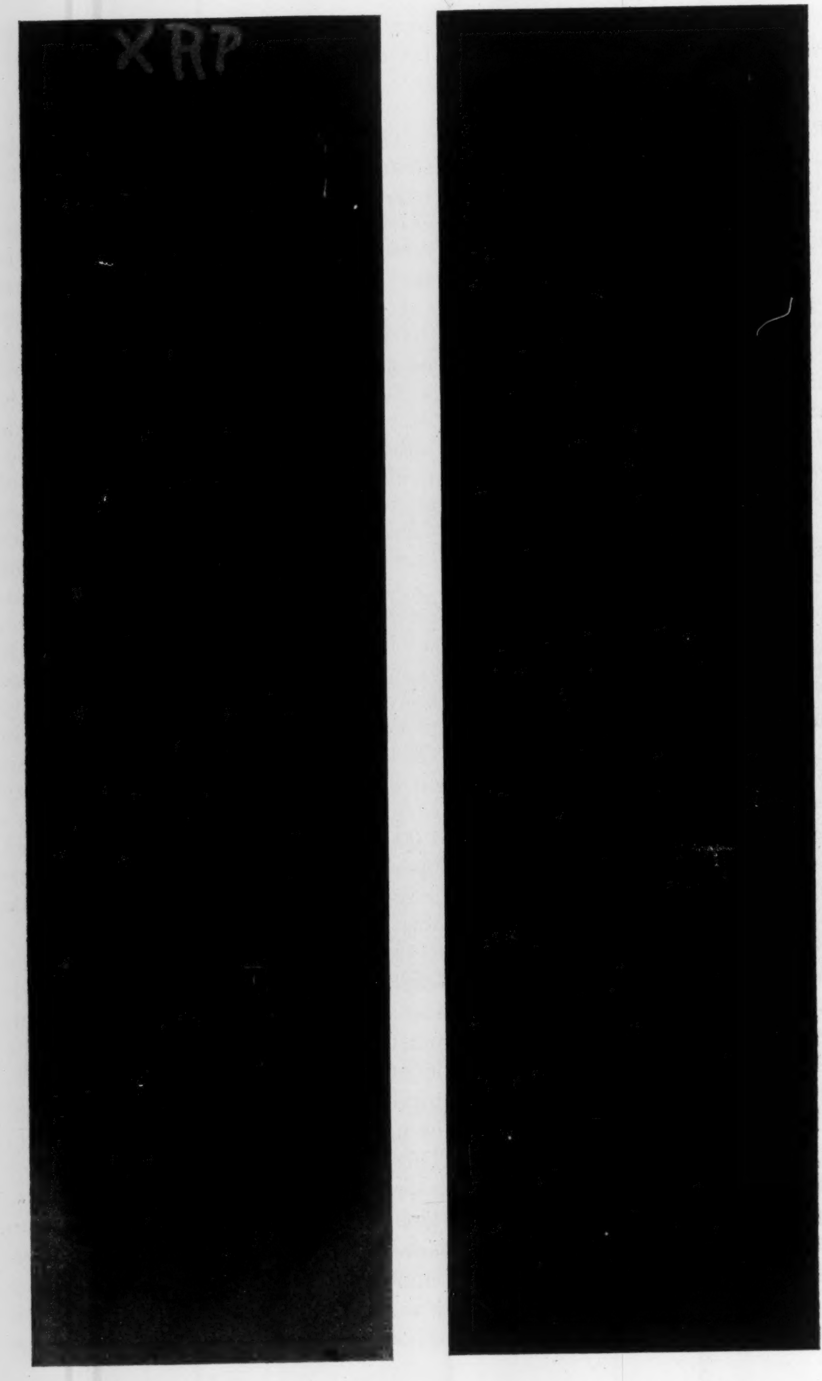
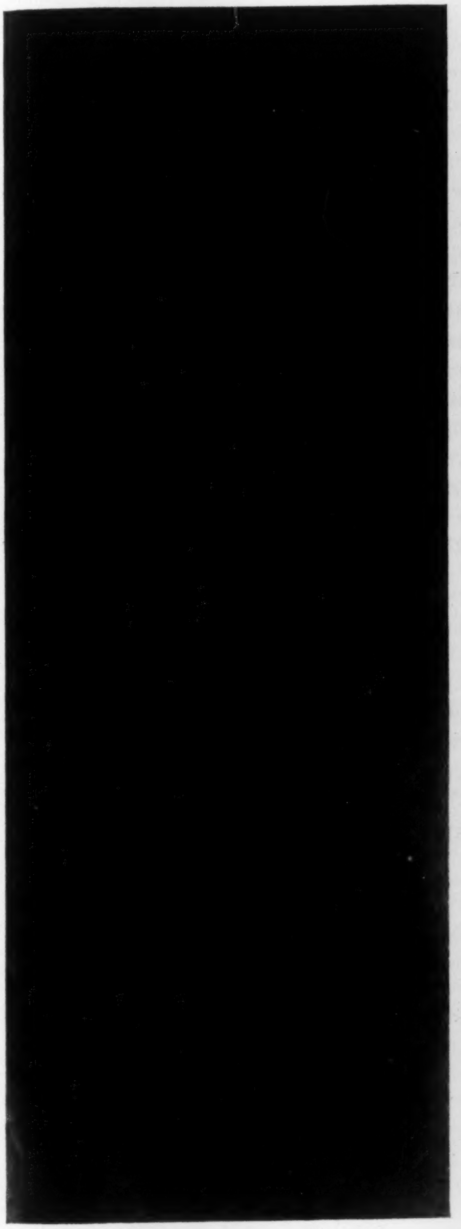


FIG. 15.—Two Exographs on 5¼ in. Thick Weld.

Top—Flexible XRP sheeting.

Bottom—Regular lead intensifying screens.



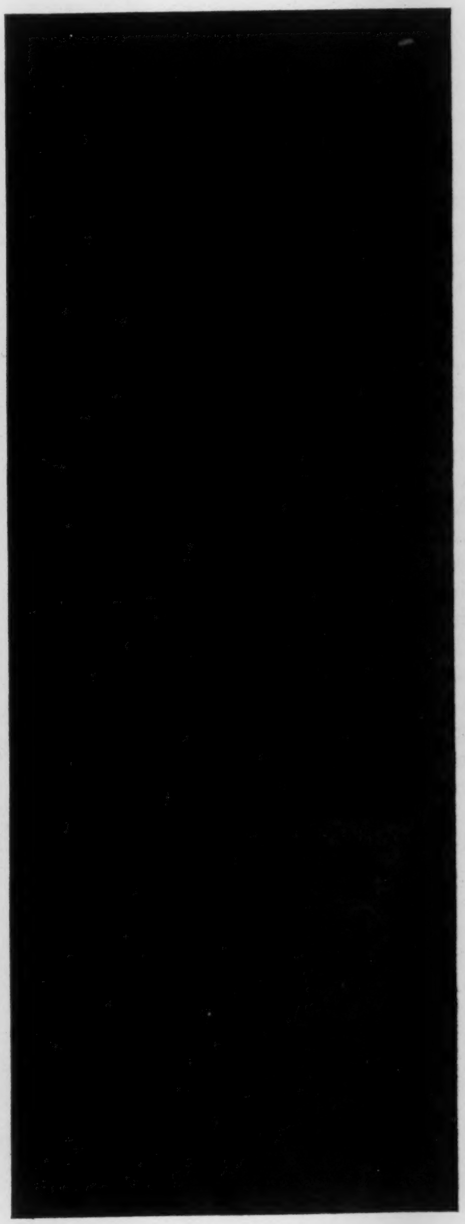


FIG. 16.—Standard Porosity Films.

No. 3.—Acceptable porosity.

No. 4.—Borderline porosity, not acceptable.

metal, the total length end to end of the inclusions would be $1\frac{1}{2}$ in., which is within the allowable length of a single inclusion. This double inclusion, however, is not allowable, since the inclusions are not separated by the requisite length of 6*L*. On light plate, acceptable porosity may occupy a good percentage of the total weld volume, but one small slag inclusion may have to be removed. The question then arises as to how to differentiate a small slag inclusion from a similar size porosity spot.

It is realized that the whole question is a difficult one to solve. The actual weld is three-dimensional; the exograph is only two-dimensional. Much work is being done, however, in an attempt to determine just how detrimental various defects are. Some manufacturers have built boiler drums and purposely added known defects and then tested the drum in fatigue above its designed working pressure until failure occurred. The indications thus far are that the weld defects are not so detrimental as hitherto supposed. When sufficient data such as these are collected, there will be a better groundwork upon which to base code requirements. Without such a basis, caution was observed as the wisest course. The lack of any serious failures has so far justified this course.

CONCLUSION

The field of industrial radiography has grown enormously. Much of the present-day speed of production has resulted from welding. This increased use of welding has necessitated a considerable increase in the number of radiographic units in use, not only in the pressure vessel field, but also for spot checks in various structural welds not required to be radiographed in their entirety. Similarly, the field of gamma-ray radiography is expanding. It is finding considerable use especially in examinations in the field. The use of the newly developed fine-grained, high contrast film has likewise improved the detail of gammagraphs. Undoubtedly, the present era will give a further impetus to industrial radiography much like that given medical radiography in World War I.

Acknowledgment:

The cooperation of E. C. Chapman, A. J. Moses, and J. N. Chatham, of Combustion Engineering Co., Inc., is gratefully appreciated. The wide experience of these men has been a considerable help in the preparation of this paper.

DISCUSSION

MR. DON M. MCCUTCHEON.[1]—The author, in mentioning X-raying girth seams, described the use of two lead filters and a screen. Just what is meant by the third screen?

MR. R. E. LORENTZ, JR.[2]—First of all, one must have a heavy filter between the film and the plate to absorb scattered radiation, that is, radiation scattered in the plate thickness. Secondly, behind the cassette holding the film, one must have another filter of lead or some other suitable material to stop radiation scattered from the room itself. Thirdly, inside the cassette one should have intensifying screens of some sort, either lead screens or some other suitable material which is flexible and can be bent around the outside of the seam.

There is the following difficulty with this type of X-raying: When the lead screens, within which the films are wrapped, are placed around the circumference of the seam, the screens wrinkle and give the films a mottled appearance because there is not a close contact at all times with the film. The lead must be backed with some suitable material such as aluminum to keep the screen from wrinkling. A different intensifying screen rather than a lead screen may also be used. The General Electric Co. has a material called XRP sheeting, which is rubber impregnated with lead, which can be used in place of the lead screens as an intensifying medium, and just as clean an exograph can be secured as with the lead screen, but with a lower intensification factor.

MR. H. H. LESTER.[3]—I should like to ask the author how he places the penetrameters when X-raying a boiler, for instance, from the inside?

MR. LORENTZ.—The penetrameters are placed on the inside, adjacent to the weld, one penetrameter mounted on each quarter. There are just four penetrameters.

MR. A. L. CHRISTENSON.[4]—I should like to ask Mr. Lorentz whether he has used the stereoscopic method for examination of welds. We have found it possible to follow beads, cracks and all various defects right down through a weld 2 or 3 in. thick in that manner.

MR. LORENTZ.—No, we have not. We have been going mainly on judgment. We know the type of welding groove which has been used, and we can generally judge from the defect approximately —not always—where that defect is, and so know where to go for removal. We have not used the stereoscopic method except for special purposes.

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[1] Metallurgical Engineer, Main Laboratory, Rouge Plant, Ford Motor Co., Dearborn, Mich.

[2] Metallurgist, Combustion Engineering Co., Inc., Chattanooga, Tenn.

[3] Principal Physicist, Watertown Arsenal, Watertown, Mass.

[4] Junior Metallurgist, Division of Physical Metallurgy, Naval Research Laboratory, Anacostia Station, Washington, D. C.

SOME CALIBRATION DATA AND SCATTER MEASUREMENTS FOR THE RADIOGRAPHY OF MAGNESIUM AIRCRAFT CASTINGS

By L. W. Ball[1]

The purpose of this paper is to provide radiographers who are already familiar with the X-ray examination of aluminum alloy castings with information required for the satisfactory examination of magnesium castings. This information consists of measurements of penetration, of sensitivity, and of scattered radiation.

Penetration data can best be represented by the two types of calibration graphs shown in Fig. 1. Figure 1(*a*) shows the kilovoltage required to penetrate a given thickness of magnesium with a selected exposure factor. Figure 1(*b*) shows the exposure factor required to penetrate a given thickness of magnesium with a selected kilovoltage. These curves show that, so far as penetration is concerned, 1.9 in. of the magnesium alloy is equivalent to 1.35 in. of aluminum, or 1.0 in. of the 4 per cent copper aluminum alloy. They also show that 55 kv.p. X-rays penetrate magnesium as easily as 68 kv.p. X-rays penetrate duralumin. From the slope of the curves in Fig. 1(*b*), it is evident that for 70-kv.p. filtered radiation, the half-value layer for magnesium is 0.60 in., whereas for aluminum the half-value layer is 0.43 in. and for duralumin it is 0.33 in.

For the radiography of material of constant thickness such as magnesium sheet, it is important to know how the sensitivity varies with the kilovoltage. Experiments were made with a block of magnesium alloy 1 in. thick and containing milled grooves 1/8 in. wide and 10, 5, 2, and 1 per cent deep in the top surface. The results shown in Fig. 2 were obtained by photometry of films each of which was exposed to a total density of 1.0.

Although the human eye can just detect a film density difference of 0.02 units under favorable conditions, for aircraft radiography it is better to use the more conservative definition of sensitivity as the difference in metal thickness that will produce a film density difference of 0.03 units. This definition has been used for Fig. 2 and for subsequent calibration curves.

For the radiography of castings in which a considerable range of thickness occurs, calibration curves similar to the type shown in Fig. 3 are essential. The vital feature of such calibration curves is that they show the range of metal thickness over which sensitivities of 1, 2, 5, and 10 per cent are attained.

No one such calibration is suitable for all the various shapes of casting patterns. As with aluminum castings and with steel castings, it is of the most fundamental importance to divide magnesium aircraft castings into three groups based on the manner and degree to which secondary radiation spoils the primary sensitivity. An appropriate calibration must be chosen for each group such that when a casting is placed in the proper group, scatter does not reduce the

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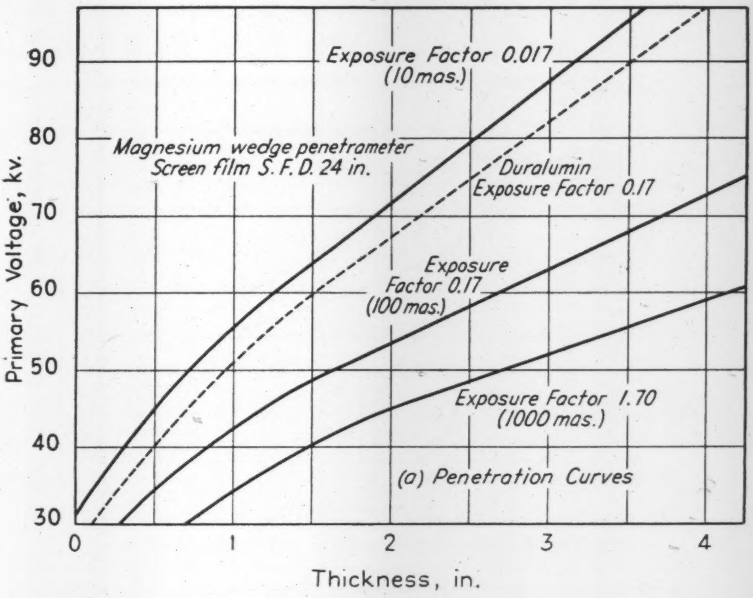
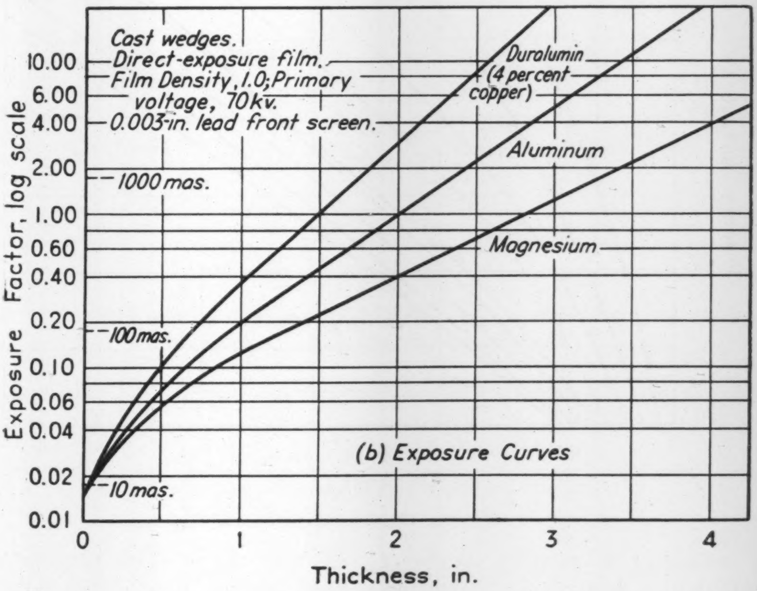
[1] Research Physicist, Triplett & Barton, Inc., Burbank, Calif. Formerly with the National Research Council of Canada.



(*a*) Penetration curves



(*b*) Exposure curves

FIG. 1.—Penetration and Exposure Curves.

claimed penetrameter sensitivity by more than twice.

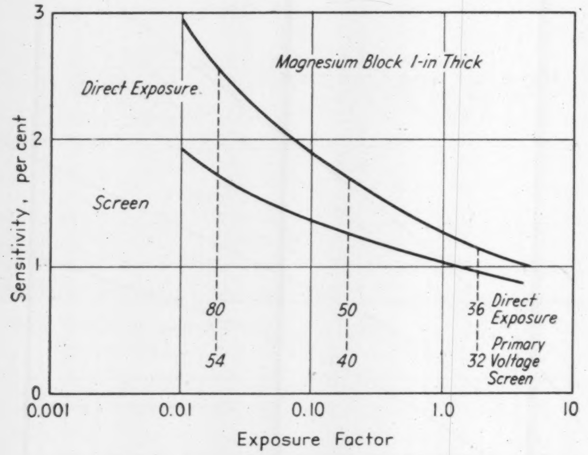


FIG. 2.—Dependence of Sensitivity on Exposure Factor.

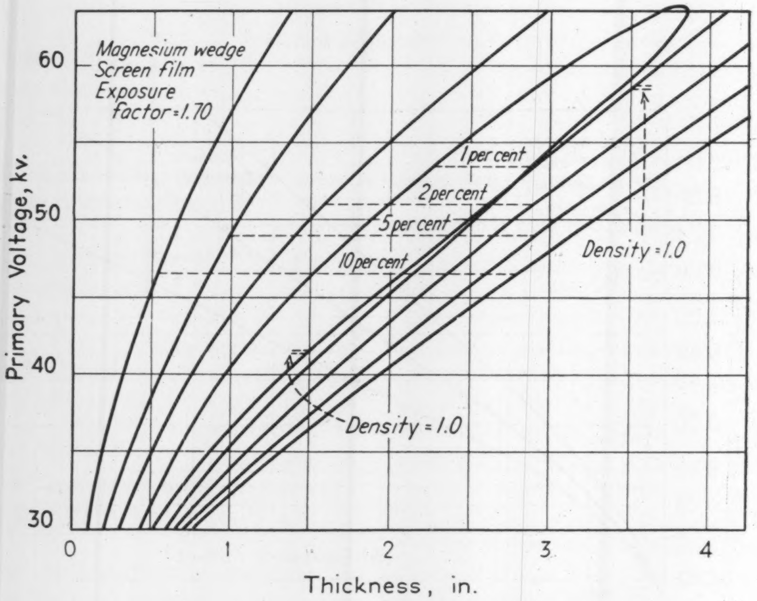


FIG. 3.—Calibration Chart of Well-Blocked Castings.

For "well-blocked" castings, the choice of a suitable calibration is straightforward. It is simply a matter of determining what exposure factor, screens, and film will give the maximum sensitivity within the practical limits of exposure time. In the radiography of aircraft castings, the practical range of exposure time, source film distance; etc., correspond to exposure factors from 0.017 to 1.70, mas. per S.F.D.^{2} and therefore, as can be seen from Fig. 2, a calibration using screen film with an exposure factor of 1.70 is suitable for "well-blocked" magnesium aircraft castings.

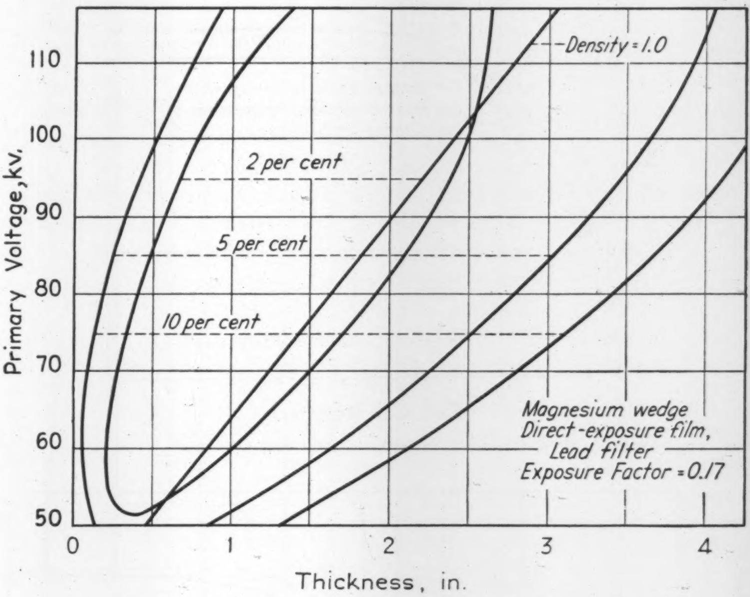


FIG. 4.—Calibration Chart for Badly Blocked Castings.

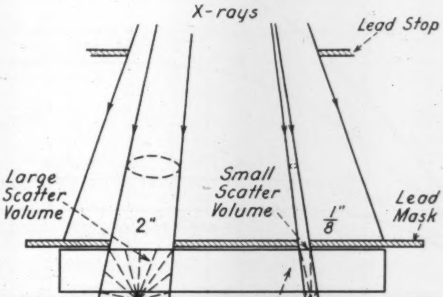

X-rays
Lead Stop
Large Scatter Volume
2"
Small Scatter Volume
1/8"
Lead Mask
P (2")
P (1/8")
Film
Magnesium Block

FIG. 5.—Scatter Measurement - Field Size Method.

For "moderately blocked" castings, the ordinary design of fluorescent cassette produces a marked loss in sensitivity at the edges of the image. This loss can be greatly reduced by using a more suitable cassette design.[2] For magnesium castings, it is simplest to eliminate the edge loss completely by using a direct exposure film in a cardboard holder with a back lead screen.

For "badly blocked" castings, the same consideration applies to the image edges, but a much more serious loss in sensitivity will occur in the images of thick sections if a calibration like that of Fig. 3 is used. For these castings, it is essential to use a relatively low exposure factor and a front filter screen. A calibration chart made with these features and suitable for "badly-blocked" magnesium castings is shown in Fig. 4.

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[2] L. W. Ball, "A Study of Cassette Design for the Radiography of Aircraft Castings," see p. 1156.

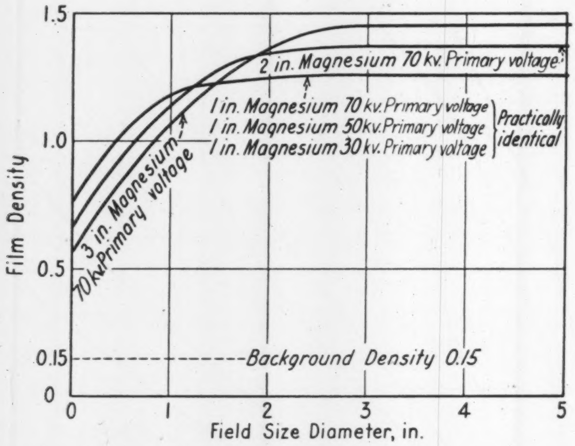

FIG. 6.—Film Density - Field Size Curves.

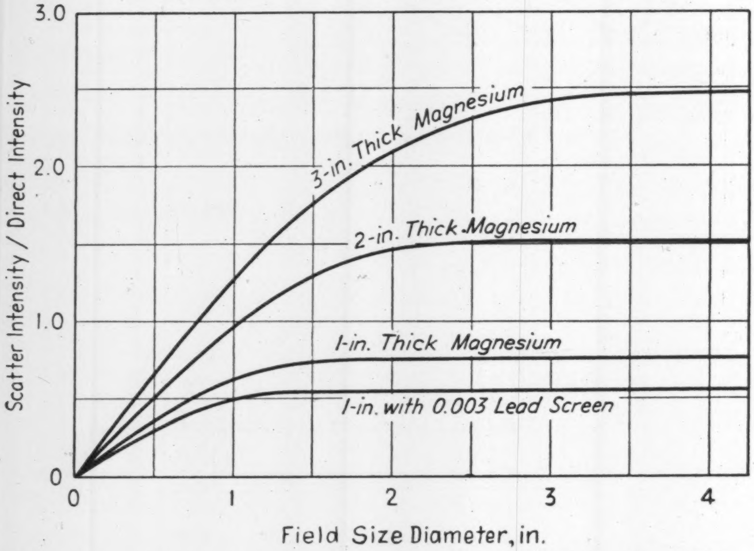

FIG. 7.—Ratio of Scatter to Transmitted Intensity (Primary Voltage, 30 to 70 kv.).

Turning now from calibration data to scatter measurements, Fig. 5 shows the experimental method described to the Society last year for the measurement of forward transmitted scatter from the casting itself.[3] This form of scatter is always present, and it is obviously important to have information about it.

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[3] L. W. Ball, "A Study of Secondary Radiation in Relation to the Radiography of Aircraft Castings," ASTM BULLETIN, No. 115, March, 1942, p. 27.

The principle of this experimental method is that the primary X-ray intensity is independent of the field size, but that the scatter intensity is not. Figure 6 shows some results obtained with magnesium sheet, and Fig. 7 presents the same results in a more usable form. These curves show that the ratio of the scatter to the transmitted intensity increases approximately linearly with thickness. It is this ratio that is important in the radiography of uniform sections.

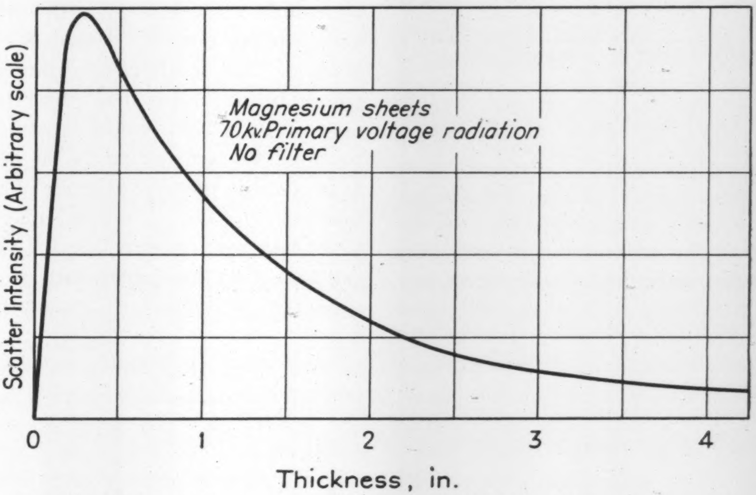

FIG. 8.—Relation of Scatter Intensity to Section Thickness (Fixed Exposure.)

For "badly blocked" castings, the image of one section can receive scatter from other sections of different thicknesses, and it is very important to know how the intensity of this scatter depends on the thickness of the section producing it.

Figure 8 shows the relation of the intensity of forward scatter to section thickness for magnesium and unfiltered 70-kv.p. radiation. It reveals the relatively deleterious effect of thin sections. The 0.003-in. front lead screen recommended for "badly blocked" castings reduces the effect of scatter from these thin sections by providing an initial filtration equivalent to 0.6 in. of magnesium.

In the radiography of aluminum castings, the lateral dimensions of all the common types of defect are sufficient to produce a fairly sharp image with any of the usual types of film and screen. But for magnesium, the prevalent microshrinkage is so fine that film grain size and intensifying screen flare are important.

To provide a precise comparison between a fine-grain direct-exposure film and ordinary screen film, a slit tapering from 0.025 in. to zero width was made in a 0.1-in. magnesium sheet, and this was radiographed underneath a 2-in. magnesium block. Photometer traces were made across the images of the slit and the results are reproduced in Fig. 9.

It is evident that with fine-grain film, the full contrast is maintained even down to a width of 0.005 in., but that with fluorescent screens, the contrast with a width of 0.005 in. is three times less that with a width of 0.020 in.

Among the several legends that have arisen in radiography is the one that magnesium produces so much more scatter than aluminum that good radiographic quality is unobtainable. Comparison of the measurements presented for magnesium in Fig. 7 with those presented previously for aluminum[3] show that weight for weight magnesium and aluminum actually produce very nearly the same amount of scatter. What then is the reason for the belief that magnesium is difficult to radiograph? One possibility is that the low kilovoltage required to compensate for the low density of magnesium has not often been available. Another possibility is that the relatively intense external scatter has been mistaken for scatter from the magnesium itself.

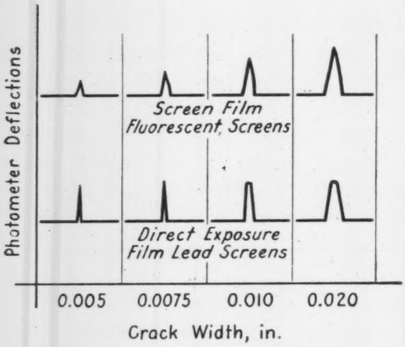

FIG. 9.—Resolving Power Test.

Direct tests of the relative sensitivity obtained with magnesium and pure aluminum show that actually they give very similar results. However, similar tests of the sensitivity obtained with common duralumin show that the presence of 4 per cent copper is sufficient to improve the sensitivity by 30 per cent. It is possible that the absence of a heavy element in the common magnesium alloy has been responsible for the supposition that low sensitivity is due to abnormal scattering by magnesium.

DISCUSSION

MR. GILBERT E. DOAN.[1]—What is meant by well-blocked and moderately blocked castings?

MR. L. W. BALL.[2]—A well-blocked casting is one in which scattered radiation produces no trouble at all. For instance, in the radiography of a large flat plate, scattered radiation cannot reach the film without going through the plate. For a well-blocked casting the sensitivity attained is just the same as with the usual type of penetrameter. This type of penetrameter is itself a flat block of metal placed on the film.

A moderately blocked casting is one in which trouble occurs only at the edges. Scatter from the cassette material is one form of trouble that is localized at the edge of the image.

A badly blocked casting is one in which scatter from thin sections undercuts into the image of thick sections and ruins the image. I have an illustration of this in the cassette paper.[3] If you have a thin shell with a thick boss on it the radiation penetrating the shell is very intense and the scatter it produces is very intense. The radiation passing through the thick boss is weakened to such an extent that the primary sensitivity is obliterated by the scatter.

MR. OTTO ZMESKAL.[4]—I should like to ask Mr. Ball what density he finds necessary in his magnesium radiographs to get maximum sensitivity. I know we are increasing that density all the time in steel work. Is a density above one necessary to get maximum reading sensitivity?

MR. BALL.—If you are dealing with a uniform thickness, the answer is that up to a density of at least two and one-half the sensitivity continually improves with a direct-exposure type of film. With the fluorescent screen type of film maximum sensitivity is reached between 1 and 1.5.

In the radiography of castings one has a variation in casting thickness and a corresponding variation in film density—dense images of thin section and light images of heavy sections. The range of thickness over which satisfactory sensitivity is attained is, therefore, very much affected by the film characteristics. In the radiography of castings a density range from 1.0 to 5.0 is satisfactory with direct exposure film and a density range from 1.0 to 3.0 is satisfactory with screen film.

MR. H. R. ISENBURGER.[5]—I should like to ask whether the author has ever tried to use a different type of radiation other than tungsten, for instance, molybdenum or even copper.

MR. BALL.—I have tried to use voltages lower than the 30 kv., which is the usual lower limit of tungsten tubes, by using tubes built for crystal structure work. I have tried molybdenum target tubes at 20 kv. Unfortunately at very low voltages fluorescent screens lose their speed and so the low-tube output necessitates very long exposures. I think that tubes with other targets than tungsten would have to be for very special jobs in which you could afford longer exposure.

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[1] Professor of Metallurgy, Lehigh University, Bethlehem, Pa.

[2] Research Physicist, Triplett & Barton, Inc., Burbank, Calif. Formerly with the National Research Council of Canada.

[3] See p. 1156.

[4] Assistant Professor of Metallurgy, Illinois Institute of Technology, Chicago, Ill.

[5] President, St. John X-ray Service, Inc., Long Island City, N. Y.

MR. ISENBURGER.—We have had occasion to use molybdenum radiation on a difficult exposure of aluminum spot welds, with very good results.

MR. BALL.—I think I could lend Mr. Isenburger some support there from microradiography. In zinc die castings we have found cavities of all sizes from $\frac{1}{8}$ in. down to microscopic limits. To study a casting completely we radiograph it to see the big flaws. Then we photograph a little section, $\frac{1}{2}$ mm. thick, using a molybdenum target tube and one of the grainless films which the Kodak Co. produces. The film is viewed under the microscope and tiny defects are revealed.

When you are pulling test bars, it is sometimes interesting to see whether the tearing takes place between the crystals or through the crystals. That is another case in which molybdenum radiation and microradiography is worth while.

MR. R. S. BUSK.[6]—Could the author amplify a little on this comparison of porosity and such things as wedges and easily seen defects? We have found considerable difference, I think, in the sensitivity when an ill-defined image is present such as porosity, and where there is a wedge.

MR. BALL.—Yes, I think there is a field there which is very important.

We regard a penetrameter as a basic calibration device. When we say the penetrameter sensitivity is 1 per cent, we do not mean that the sensitivity for microporosity is 1 per cent. The sensitivity for microporosity depends on the human eye, the viewing lamp and several other things. But from experience we say: "Here is a test bar with an acceptable degree of microporosity in it. A technique with 1 per cent penetrameter sensitivity showed this condition, so we are safe in using the technique."

MR. BUSK.—I wonder also whether you could expand a little on the use of screened films. Do you recommend them?

MR. BALL.—There is an historical factor there. Before the war started, we had to persuade foundries to use X-rays. It was much easier to persuade them to spend $1200 on a little tube than it was to spend $5000 on a high-voltage tube, so we recommended that they get a $1200 tube, 80 kv., and use fluorescent screens to get sufficient penetration. They had to go to a lot of trouble with blocking and the use of carbon tetrachloride, but they did get results. So historically we started off with fluorescent screens to bridge the gap between $1200 and $5000.

Today these screen films are restricted to the radiography of thick uniform sections. For a fixed exposure time, fluorescent screens and low voltage give more contrast but less sharpness than direct exposure and high voltage.

MR. DON M. MCCUTCHEON.[7]—With regard to penetrameters, there are innumerable cases where you can show to best advantage either the penetrameter or the casting. If the specifications or the people who are supporting the penetrameters realized that, we would all be better off. We have a large number of castings where we can improve the clarity of the image by proper filtration at the tube or at the film, at the expense of the penetrameter image. This filtration degrades the penetrameter quite considerably, but improves the image of the casting. The improvement obtained by putting the filter on the tube is not quite as great as the improvement by putting it on the film, but it is sometimes more convenient.

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[6] Metallurgist, The Dow Chemical Co., Midland, Mich.

[7] Metallurgical Engineer, Main Laboratory, Rouge Plant, Ford Motor Co., Dearborn, Mich.

MR. H. H. LESTER.[8]—The question of the penetrameters comes up, and I should like to ask the author whether he is regarding the penetrameter as a sensitivity gage.

MR. BALL.—We regard it very definitely as a basis for sensitivity and as a penetration gage, but we have to go beyond that and make allowance for scattered radiation, etc.

MR. LESTER.—I should like to point out that there are two concepts of the penetrameter. One is the concept that the author is illustrating, where the penetrameter is definitely a sensitivity gage. There is another, however. I think the Boiler Code penetrameter illustrates it. The penetrameter that the Boiler Code deals with consists of a flat steel plate with three holes drilled in it, and that penetrameter is placed on the outside of the metal adjacent to the source of radiation. If the penetrameter images show sharply—I mean by that, that the edges are clearcut, sharply defined—then the image of any defect which is more remote from the source of radiation, and therefore has a more favorable d/t (source-cavity to cavity-film distance) ratio, should show theoretically with greater clarity. Of course there would not be any appreciable difference, so that this penetrameter indicates principally whether the working distance of the tube is correct. It will give the working conditions under which sharp images may be expected and that is about all that it will do.

The use of the penetrameter as a sensitivity gage is another thing altogether. It seems to come up more in the radiography of aluminum castings, and I have been impressed with the fact that in the radiography of aluminum castings radiography seems to be becoming more and more a quantitative tool. In the radiography of steel castings, to which we have been accustomed, the radiographic picture is rather qualitative than quantitative. We see the image which indicates the presence of the defect, but we do not always measure the extent of that defect because we do not see all of the image, and we do not make the quantitative measurements which the author has been illustrating. I think, however, that the aluminum radiography is pointing the way for a better use of radiography in steel.

MR. B. A. SILARD.[9]—Is it not true that when we look for sharpness of the image we have in mind that we want to see the image by eye and we could possibly put up with less sharpness if we would confine ourselves to direct density measurements?

Let us take an example: We have a flaw somewhere in the metal and we forget about locating and identifying it by eye. The eye, of course, is not too good in making out differences when they are gradual from one side of the picture toward the center of the flaw. However, if we measure the density difference between the edge of the image and the spot where the flaw is supposed to be, we could put up with a considerably blurred image, and I would like you to discuss the measurements by densitometer *versus* visual comparison.

MR. BALL.—That is an important question which I think I can be specific about. With coarse-grained film the photometer is no more sensitive than the eye for observing sharp images because when you take a photometer reading of the film you get a background density that oscillates as you go through the grains. You are looking for a defect, which should give a variation in the photometer trace similar to the variations in background density. So sensitive photometry is dependent on the use of fine-grained films. With fine-grained film, photometry offers a chance of seeing things which the eye cannot.

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[8] Principal Physicist, Watertown Arsenal, Watertown, Mass.

[9] Photovolt Corp., New York, N. Y.

MR. SILARD.—Would you restrict this to microporosity, or does this hold true for large flaws?

MR. BALL.—I think that in the case of radium radiography photometric analysis is most likely to be useful. For example, say you have 16-in. armor plate and you put radium on one side and the film on the other. The images of the defects will not be sharply defined; they will consist of diffuse variations in density. I think that for such blurred images, even a simple photometer would be superior to the eye, providing you are radiographing something like a flat plate where your background is easily interpreted.

MR. SILARD.—Another question is this: What are the minimum densities we are attempting to get? Or, to put it in a different way, with regard to the minimum densities we want to get, are they determined by the shortcomings of the eye or are they limited by the sensitivity of densitometers, and what are, actually, those densities? We have seen 2, 2.5, and higher. Are those actually necessitated?

MR. BALL.—In the radiography of nonuniform subjects the density is one part of the jigsaw. What you are trying to get out at the end is a certain sensitivity. For example, the 2 per cent sensitivity may correspond to densities of 0.6 to 5.0. These densities are incidental to the fact that this is your 2 per cent range.

We use a densitometer many times a day. For instance, a film comes in from an outside laboratory with a light-looking spot on it. We may think that it is under-exposed. We measure it and if we find a density of 0.2 we are certain it is under-exposed. If we find a density of 0.8, we know that it is an optical illusion, probably due to an adjacent dense area which made it look under-exposed.

MR. SILARD.—My third question: Do you feel that it will always be necessary to include the step of photographing, or is there hope that work can be speeded up by direct photoelectric or other photometric measurement on the fluorescent screen or otherwise?

MR. BALL.—For castings I think photography is the ideal method. I cannot even conceive of anything replacing photography. You have in the aircraft industry a certain amount of legal responsibility. The man in the plane is absolutely dependent upon the men who put that plane together, and there are just no limits to the trouble to which we will go. If a plane crashes and it was obvious that some part failed, you are going to go back into your records. You are going to find the radiograph of every casting of that pattern, find what was the prevalent type of defect. If your records are good you will locate the individual casting.

MR. SILARD.—Would this refer to more uniform cross-sections, to armor plate and things of that kind, a strip of metal or to a welded seam?

MR. BALL.—I think in both those cases electronic methods offer something. There are certain inherent defects in forgings in which a wavy background is what you get out of a radiograph. To the untutored eye it just looks like a bad development, but to the tutored eye it is a particular state of that forging which is dangerous, and the eye makes a qualitative measurement. It should be possible to use photometry and to find some device for saying what is good or bad, in forgings.

MR. MCCUTCHEON.—There is one reason why electrical methods may not be applicable to this type of work. The author stated the effect of grain on the photometry of films. There is another effect, namely, that all castings have variations in thickness which cannot be controlled within too narrow limits, and anybody who has made a photometer curve of a film which to the eye was perfectly uniform in density, will know that the curve will go up and down because of differences in thickness of the casting.

But there is one possibility of electric detection which might eliminate the film, namely, in the case of large steel castings, where the foundry method is worked out satisfactorily from the foundry's standpoint, eliminating shrink and so forth. There are always things that cannot be too closely controlled in the foundry process—temperature of pouring, temperature of melting, and such things—that are liable to produce hot tears in changes of section. Here electric recording might find an application eliminating films.

MR. STUART V. CUTHBERT.[10]—In the production X-raying of magnesium castings, we have found that such problems of porosity or inclusions or shrinkage are secondary to microshrinkage. We have also found through an error that in X-raying magnesium castings, using the film recommended for the million-volt machine and an exposure time from seven to eight times the normal time used on a standard no-screen film we will get sharper definition and greater contrast—surprising results.

MR. BALL.—I think that these new films are undoubtedly suitable for magnesium castings.

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[10] Supervisor of X-ray, Wright Aeronautical Corp., Paterson, N. J.

A CORRELATION OF THE MECHANICAL PROPERTIES AND RADIOGRAPHIC APPEARANCE OF MAGNESIUM ALLOY CASTINGS

By R. S. Busk[1]

Synopsis

A method for the quantitative rating of radiographs showing porosity in magnesium base alloys is described. By varying the intensity of the viewing light with a Variac, and noting the voltage at which porosity is just visible, a scale is established for the rating of radiographs. This scale is then used to study the effect of microporosity on the tensile properties of Dowmetal H alloy in the as-cast and solution heat treated states; the tensile properties of C alloy in the as-cast, solution treated and heat treated and aged states; and the fatigue strength of C alloy in the solution heat treated state.

It is noted that C alloy will be less porous than H alloy under similar foundry conditions. But with a given amount of porosity H alloy will suffer a smaller loss of strength than C alloy. The tensile strength of both alloys falls as the amount of porosity increases, but the yield strength is unaffected until very large amounts of porosity are present. The fatigue strength of solution heat treated C alloy is affected to the same degree as is the tensile strength for a given amount of porosity.

Since radiography is being used more and more in the metallurgical industry as an inspection tool, the problem of evaluating the appearance of the radiograph of a metal casting is becoming increasingly important. The present study was undertaken to test a method of quantitative impersonal evaluation of a radiograph as well as to investigate any correlation that might exist between the radiographs of magnesium alloy castings and the mechanical properties of those castings.

Experimental Technique

Appearance of the Radiograph:

Slag or flux inclusions, gassy metal, or oxide inclusions seldom occur in magnesium alloy castings. A defect that occurs occasionally is micro-shrinkage, also known as micro-porosity, shrinkage porosity, or porosity. Therefore, attention was directed toward a correlation of the effect of porosity, as evaluated by its radiographic appearance, with the mechanical properties. Micro-shrinkage exists as small intergranular holes of irregular shape. They are usually of the order of 0.001 to 0.005 in. long, and tend to cluster in a small volume of metal. It is generally believed that they are due to lack of proper feeding while the molten metal is freezing and contracting. On the radiograph the individual holes are not visible, but the various clusters appear on the film as vaguely outlined, wavy lines, or areas of greater density of blackening than the surrounding areas on the film. The contrast between the density of blackening due to the porous areas and that due to the sound metal increases as the amount of porosity increases.

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[1] Metallurgist, The Dow Chemical Co., Midland, Mich.

Quantitative Evaluation of the Radiograph:

It was found that for a given contrast on the film there was a certain minimum of viewing light intensity required to see that contrast. This minimum decreases as the contrast increases. Therefore, by varying the intensity of the viewing light, different minima will be found corresponding to different amounts of contrast on the film. A voltage Variac[2] was connected in series with a standard General Electric viewing screen, containing a 75-watt blue frosted Mazda bulb. By varying the voltage across the light, the intensity transmitted through the film was also varied. For a given film contrast a light intensity just sufficient to make visible the contrast was found at a given voltage. Later work showed that the reciprocal of the voltage gave a better curve with the property data. For ease of handling, the numerical values of the reciprocal are multiplied by 10,000. This number is known as the "contrast index" and has the symbol 1/v. Thus the greater the contrast on the film, the greater will be its contrast index.

The method, then, consists in placing the radiograph on the viewing screen with the intensity of the light equal to zero. The intensity is gradually increased until porosity is just visible on the radiograph. The voltage reading on the Variac is recorded as a figure representing the contrast on the film. Comparison of the contrast index, for a radiograph of a cast section showing porosity with curves established as shown below, gives an estimation of the tensile strength of the section.

To check the validity of the assumption that the Variac voltage reading will correctly distinguish between areas of different contrast, a series of test specimens were made. These consisted of blocks of constant thickness with holes of rectangular cross-section and variable thickness running lengthwise through the center. The blocks were radiographed in a direction normal to the thickness of the holes, and the voltage reading at which each hole could be just seen was recorded. The results, as shown in Fig. 1, indicate that a logical relation exists between the voltage reading and film contrast. Although the point was not plotted, it was found that a void of 0.003 in. could be seen in a piece 0.400 in thick. Therefore, under these ideal conditions, with a large hole having sharply outlined edges a defect amounting to 0.75 per cent of the total thickness can be seen. The sensitivity is not so great as this when the vague shadows produced by porosity are being examined. The effect of total thickness of the piece is also shown on these curves. It will be noted that as the thickness increases the voltage required to just see a given percentage void de-

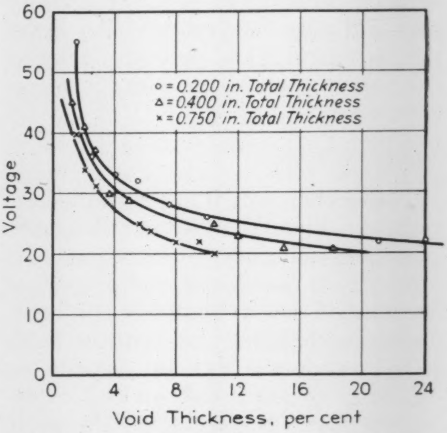

FIG. 1.—Effect of Void Thickness on Radiographic Contrast.

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[2] Type 200C, manufactured by General Radio Co., Cambridge, Mass.

creases, that is, the contrast increases. Although this indicates the necessity of determining the effect of thickness on a comparison of the mechanical properties of test bars and the amount of porosity, subsequent work has shown that the effect is not large, and in practice may be disregarded in thicknesses below 1 in.

Further work with the test blocks showed that a background of 1.0 to 1.2 density units[3] gave the best results. This density seems to produce a greater difference in contrast, as here measured, between differing amounts of porosity than will a heavier density. For quantitative distinction between differing amounts of porosity this point is important.

Sources of Error:

The errors involved in the method are twofold. First the different radiographs must be uniformly exposed and developed. It was found that a range of background density from 1.0 to 1.2 produced no change in the contrast index if the range was due to variation in exposure technique and not in developing the film. A very small change in background density due to developing technique produces a relatively large change in the contrast index. Consequently, this technique was held closely to the procedure recommended by the Eastman Kodak Co. for their Kodalk developer. The variations in exposure technique were kept to a minimum, but were great enough to produce background densities varying from 1.0 to 1.1 density units.

The second source of error lies in the viewing apparatus and in the operator judging the radiograph. It was found that the voltage readings on given radiographs were reproducible to ± 1 v., either by the same operator on different days or by different operators. Since a change in the voltage emissivity curve for the bulb will introduce an error, the relation should be checked occasionally. After 1 yr. the viewing assembly still gave the same voltage reading, within the ± 1 v. error, as was first obtained on a series of radiographs. Thus the total error in evaluating the porosity on a film is about ± 1 v.

TABLE I.—A LIST OF THE NOMINAL COMPOSITIONS AND HEAT TREATMENTS OF THE ALLOYS USED.

Alloy	Composition by Weight, per cent			Solution Heat Treatment
	Aluminum	Zinc	Manganese	
C..	9	2	0.1	Started at 500 F. Furnace raised to 760 F. and held 20 hr. Air cooled.
H...	6	3	0.2	Started at 500 F. Furnace raised to 730 F. and held 12 hr. Air cooled.

TABLE II.—THE RADIOGRAPHIC TECHNIQUE USED FOR THE 0.3 IN. THICK TEST BARS.

Alloy	Kilovolts	Milliamperes	Seconds	Film to Specimen Distance, ft.
C......	40	10	60	3
H......	45	10	35	3

Procedure:

The correlation between the static tension properties and the radiographic porosity, as measured with the Variac, was determined with Dowmetal C and H alloys, both as cast and solution heat treated. Table I lists the compositions and heat treatments. The radiography was carried out with the use of a General Electric X-ray unit, model No. KX3. The technique which was used is shown in Table II.

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[3] Density, γ, is defined as the logarithm of the ratio of the incident to the transmitted light intensity. It was measured with a Gaertner Densitometer, in porosity-free areas of the radiographs.

Test specimens were obtained by machining from sand-cast plates, 10 in. long by 5 in. wide by $\frac{1}{4}$, $\frac{3}{8}$, $\frac{1}{2}$, $\frac{3}{4}$, 1, or $1\frac{1}{2}$ in. thick. The plates were cast flat, with the sprue and gate at one end and risers at both ends. Five different risers, labeled *A*, *B*, *C*, *D*, and *E* in order of increasing size were used; *A*, *B*, and *C* on the $\frac{1}{4}$ and $\frac{3}{8}$ in. thick plates; *B*, *C*, and *D* on the $\frac{1}{2}$ and $\frac{3}{4}$ in. thick plates; and *C*, *D*, and *E* on the 1 and $1\frac{1}{2}$ in. thick plates. In this way it was possible to obtain plates with differing degrees of porosity. In the case of C alloy it was difficult to obtain large amounts of porosity with any of the above combinations, and therefore a few plates were cast without risers. Plates having no porosity were produced for each alloy. It was also possible to obtain a range of micro-shrinkage, including plates with an extreme amount of porosity, the range studied exceeding that obtained in commercial foundry practice. The solution heat treatment for each alloy was carried out in an evacuated electrically heated furnace. The test specimens were cut from sections which showed the desired amount and distribution of porosity on radiographs of the plates. The final test specimens were 5 in. long by 0.3 in. thick with a 2-in. gage length, and a 0.75 in. wide reduced section, and were radiographed through both thicknesses of the reduced section. They were then polished with No. 120 emery paper, care being taken to retain flatness and squareness. This polishing removed all machining marks. Data were also obtained on test specimens cut from commercial castings of C alloy in the heat-treated and aged condition. Since most of the bars were too short for a 2-in. gage length, no yield strength or percentage elongation figures were obtained. Although fewer test specimens were used in this case than in the other work reported, it is believed the results are valid.

The tension tests were made on an Amsler testing machine, the yield strength being taken as the stress at 0.2 per cent deviation from the modulus line. The place of fracture was noted on the radiograph of each bar and the 1/v value of the porosity at that point compared with the tensile properties.

TEST RESULTS

Tendency Toward Porosity:

The variation in foundry technique for production of the plates used permitted a comparison of the relative tendencies toward porosity of C and H alloys. If the feeding system were sufficient to make the H alloy plates radiographically sound, the C alloy plates were also radiographically sound. But with the minimum feeding system to produce radiographically sound C alloy plates, some porosity was observed in the radiograph of the H alloy plates. When the feeding system was sufficiently poor to result in C alloy being porous, the H alloy plates had a still higher degree of porosity.

Tension Tests:

The data obtained in the tension tests are given in Table III, and the tensile strengths are plotted in Figs. 2, 3, and 4. Each point represents the average of several bars.

A value of 200 for 1/v represents porosity just visible on the radiograph. When a radiograph is viewed in the normal manner, that amount of porosity which would be considered very heavy would, in the system used here, have a contrast index of about 330; that which is quite heavy, 280; and that which is light, 220 or less. Commercial castings will usually have no place with an index

TABLE III.—THE AVERAGE TENSILE DATA OBTAINED FOR C AND H ALLOYS FOR VARIOUS 1/v VALUES.

Alloy	Heat Treatment[a]	1/v	Yield Strength, psi.	Tensile Strength, psi.	Elongation, per cent
H......	S.C.	227	13 700	25 500	4.0
		250	12 600	24 500	3.5
		267	13 100	24 900	3.0
		270	12 100	22 500	2.5
		278	11 700	22 300	2.0
		286	12 900	23 600	2.0
		294	12 600	22 100	2.0
		303	12 100	22 400	2.0
		308	11 500	20 800	1.5
		313	12 400	20 100	1.5
		333	11 900	18 400	1.0
		345	11 000	18 800	1.0
	H.T.	200	13 600	37 500	10.5
		204	13 100	30 800	7.0
		233	13 200	30 800	6.5
		238	......	33 300	6.5
		250	......	31 000	5.0
		270	13 200	26 300	4.5
		286	13 500	26 600	3.5
		294	13 100	25 400	4.0
		303	13 600	27 200	3.5
		313	13 100	25 900	3.0
		323	10 400	24 300	2.5
		333	11 800	20 500	2.0
		345	10 000	22 000	2.0
C......	S.C.	200	14 600	19 000	0.5
		217	14 800	18 200	0.5
		238	14 200	18 100	1.0
		244	14 600	19 400	1.5
		263	14 100	18 200	0.5
		270	15 000	18 700	0.5
		286	13 700	17 700	0.5
		294	14 400	17 200	0.5
		303	14 200	17 500	0.5
		313	13 700	16 000	0.5
		323	13 400	15 800	0.5
		333	12 900	16 000	0.5
		345	12 000	14 400	0.5
		357	11 300	13 800	0.5
	H.T.	200	13 700	27 700	5.0
		203	14 600	29 300	5.3
		208	14 400	27 800	4.5
		218	14 200	29 000	5.0
		222	14 300	25 900	3.7
		228	14 900	26 800	3.5
		233	14 200	25 700	3.5
		238	15 500	24 800	3 0
		270	15 500	25 000	2.0
		278	16 200	25 700	1.0
		286	17 600	24 700	1.0
		294	16 200	24 500	1.0
		303	15 600	25 000	1.5
		313	15 700	22 600	1.0
		323	14 500	22 900	1.5
		333	13 700	18 700	1.5
		345	12 600	18 300	1.0
		257	10 400	14 900	1.0
		370	10 700	13 900	1.0
	H.T. A.[b]	200	......	35 400	....
		222	......	31 000	....
		238	......	31 900	....
		263	......	22 000	....
		270	......	26 700	....
		278	......	25 700	....
		286	......	29 100	....
		294	......	26 700	....
		303	......	25 000	....
		313	......	24 700	....
		358	......	17 700	....

[a] S.C. = as sand cast.
H.T. = solution heat treated.
H.T.A. = solution heat treated and aged.

[b] These results are obtained on bars cut from commercial castings. The data are based on fewer test results than is the case for the rest of Table III.

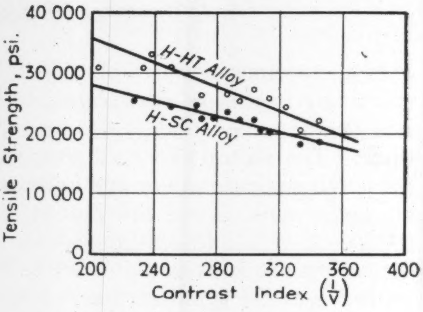

FIG. 2.—Effect of Porosity on the Tensile Strength. Bar thickness = 0.300 in.

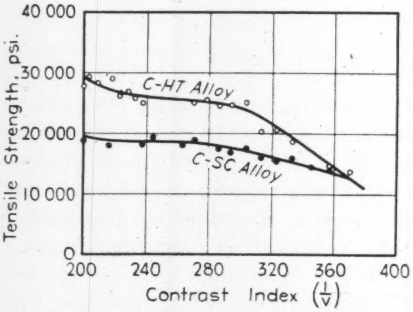

FIG. 3.—Effect of Porosity on the Tensile Strength. Bar thickness = 0.300 in.

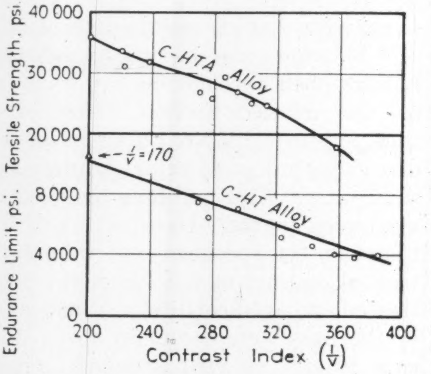

FIG. 4.—Effect of Porosity on Tensile Strength, Bar Thickness = 0.300 in., and on Endurance Limit Based on 100,000,000 reversals.

greater than 280 although sometimes they may have local areas with porosity giving a contrast index as high as 300. In general, those areas which show porosity in some commercial castings will have porosity ranging from contrast index values of 200 to 280.

These data show that a correlation does exist between the amount of porosity and the tensile strength. It was found that the tensile strength decreases as the 1/v rating increases. When the error of ± 1 v. is applied to the curves it becomes a variation of about ± 2000 psi. tensile strength. The average error of the points for each curve is found to be within these limits, and 90 per cent of the individual points also fall within the same limits.

The yield strength does not decrease appreciably until the micro-shrinkage reaches a 1/v value of 310 or more. Since this value is well above what is experienced in commercial castings, no loss of yield strength with porosity will be observed with magnesium alloys.

Effect of Variation in Thickness of the Radiographed Metal:

Since all of the preceding results were obtained on one thickness, as-cast C alloy was used to determine the effect of thickness on the correlation. Bars 0.2 in. thick with a 0.5 in. wide reduced section were prepared in the same manner as the ones previously described. The l/v values for the 0.75-in. thickness on the original as-cast C alloy bars were correlated with the tensile strengths. The values of 1/v for as-cast alloy C 0.2 in. and 0.75 in. thick are plotted in Fig. 5. The values for 0.3 in. are given in Fig. 3.

According to these curves, the tensile strength, for any given contrast index, will be lower as the thickness of the section being measured increases. However, the differences become vanishingly small as the thickness approaches 1 in. Consideration of the error of ± 2000 psi. shows that there is no difference between the 0.200-in. and the 0.75-in. thickness. Therefore, any results based on the curve for 0.300 in. will be approximately correct for any other thickness between 0.2 and 1 in.

Effect of Variation in Width of Porous Areas:

The 1/v value accounts for the depth of the porosity through the thickness of the section. Unless the piece can be radiographed at right angles to the original direction, there is no figure representing the amount of porosity across the width of the bar. This can be obtained by considering the area of the radiograph which contains porosity. A number of as-cast C alloy bars were prepared with varying amounts of porosity across the width. Upon being compared with the curve in Fig. 3 the results of these particular bars showed that when only 10 per cent of the radiograph of the test specimen is covered with porosity, the tensile strength will be 2600 psi. higher than the average for 100 per cent covered; for 15 per cent, 1800 psi.; for 30 per cent, 1600 psi.; and for 50 per

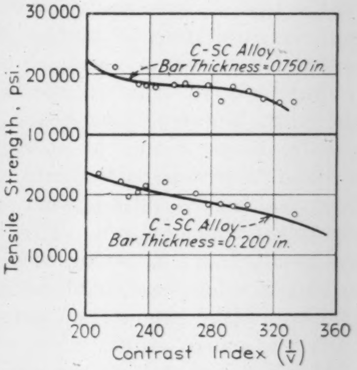

FIG. 5.—Effect of Porosity on the Tensile Strength.

cent, 500 psi. Consideration of both the depth and extent is necessary when evaluating the porosity shown in any section. The data show that a region of micro-shrinkage will uniformly affect twice the area it occupies on the radiograph. It is only when it occupies less than 50 per cent of the area that the 1/v value must be corrected.

Fatigue Tests:

In addition to the tension tests, the fatigue strength in bending of alloy C-HT was determined for varying amounts of porosity. About 150 fatigue specimens were cut from the same type of plates that have already been described for the tension tests. They were round bars, 0.3 in. in diameter at the reduced section, standard specimens for the R. R. Moore rotating beam type fatigue machine. Since the specimens were round, they were packed in Aloxite—about 600 mesh Al_2O_3 powder—and radiographed while packed. This procedure resulted in "flattening out" the picture of each bar, and enabled a good estimation of the amount of porosity through the entire cross-section. The 1/v value for each bar at the reduced section was recorded and the bars then separated into 10 groups, ranging from very heavy amounts of porosity to none visible on the radiograph. All the bars of each group (about eight in number) were radiographically homogeneous and thus could be used to determine the fatigue strength for each degree of porosity represented. The reduced section of each bar was uniformly porous.

The results, based on 100,000,000 reversals, are given in Fig. 4. The individual fatigue curves were almost flat at 50,000,000 reversals. For porous bars of C-HT, alloy 100,000,000 reversals seemed sufficient. It can be seen that a correlation exists between the amount of porosity and the fatigue strength in bending of solution heat-treated C alloy.

DISCUSSION

In each case studied the smooth curve giving the smallest average error was drawn through the experimental points. For H alloy this curve is a straight line, while for C alloy it is not. In every case either a straight line or a curved one can be drawn without increasing the average deviation above ± 2000 psi.

Since a 1/v value of 200 represents an amount of porosity just visible on the radiograph, the strength at this point is not that of sound metal. There must be a certain minimum of porosity present before the radiograph can detect it. Most areas of commercial castings have porosity which does not exceed this minimum and hence the strength values will lie between the strength of separately cast, standard A.S.T.M. test specimens and that corresponding to a 1/v value of 200. For H-HT alloy this range is from 35,000 to 40,000 psi. Also for C-HTA alloy the range is from 35,000 to 40,000 psi. For C-HT alloy, however, it is from 30,000 to 40,000 psi.

The fatigue strength of C-HT alloy is affected by porosity to about the same degree as is the tensile strength. If 14,000 psi. is taken as the fatigue strength of sound C-HT alloy and 40,000 psi. as its tensile strength, then at a porosity of $1/v = 240$ both the fatigue and tensile strengths have dropped about 35 per cent.

All of the work reported in the present paper has been done on test specimens machined flat and to accurate dimensions. Under these ideal conditions the average error in estimation of the tensile strength is ± 2000 psi. When commercial castings are radiographed it is more difficult to obtain uniform negatives. If the casting is such that the background density of radiograph negatives made from it can be kept between 1.0 and 1.2 the average error will remain the same. The greater the departure from these limits the greater will be the error.

Some work has been done in this laboratory on the problem of the application of the curves obtained from test data to the estimation of the strength of commercial castings. Flat test specimens were cut from a number of different castings of variable complexity. When the bars were radiographed, the estimation of the tensile strengths agreed with the values obtained by pulling the test specimens. It is therefore considered valid to estimate the strength of a cast section, provided the background density of a radiograph of that section can be held between 1.0 and 1.2 density units. Although the mechanical properties of sections of a casting can be estimated, no prediction of service life can be made for any casting unless the stresses to which the casting is to be submitted are known, both in quantity and in distribution.

When choosing between C and H alloys the problems of the individual casting must be considered. If the choice is simply one of greater soundness or pressure tightness C alloy should be used. However, if the choice rests with the strength that can be developed in the final casting, then two factors must be studied for each alloy. First, it must be known how much porosity will appear in the casting, and where it will appear for each alloy. Second, the affect of the amount of porosity appearing in each alloy on the strength of that alloy and thus on its service capabilities must be estimated. Since C alloy is more easily made porosity free than H alloy, and yet the strength of H alloy is less affected by porosity than that of C alloy, each casting has to be considered as a special case.

SUMMARY

1. The quantitative method of rating porosity herein described and as shown in radiographs of magnesium alloy castings gives a rating, known as the "contrast index," denoted by the symbol 1/v, having values from about 200 at just detectable porosity to values above 400.
2. The tensile strength of magnesium alloys decreases as the amount of porosity increases.
3. A 1/v rating of 300, representing an amount of porosity that is heavier than usually obtained in the foundry, represents a tensile strength of solution-heat-treated H alloy within the specified minimum for bars cut from castings.
4. The yield strength of magnesium alloy castings is very little affected by porosity.
5. The fatigue strength of solution-heat-treated C alloy also decreases with increasing porosity.
6. C alloy has a lesser tendency to develop porosity than H alloy.
7. The mechanical properties of H alloy are less sensitive to porosity than are those of C alloy.
8. The alloys are more sensitive to porosity in the solution-heat-treated than in the as-cast state.
9. The fatigue strength and the tensile strength of solution-heated-treated C alloy are about equally sensitive to porosity.

DISCUSSION

Mr. L. W. Ball[1] (*by letter*).—The results of this research provide information of the type most urgently needed for intelligent interpretation of radiographs. The author's proof that strength reduction in cast metal depends on alloy composition as well as on radiographic appearance emphasizes the fact that radiographs should be interpreted only by fully trained specialists.

In the discussion, no reference is made to the methods used to reduce variations in personal vision. Was a routine employed for accommodating the observer's eyes?

Is the recommended film density of 1.0 to 1.2 based on the minimum illumination required for detecting microporosity, or on the sensitivity obtained with a constant transmitted brightness?

Is the effect of section thickness shown in Fig. 5 based on using the same kilovoltage for all thicknesses, or on using a constant exposure factor? The contrast index for a given thickness will depend on the effective kilovoltage, and it might be possible to obtain coincident curves by using a constant exposure factor.

For specimens which were not uniformly porous, was the contrast between sound and unsound regions taken into account, or just the contrast within the porous region?

Mr. R. S. Busk.[2]—Before making any readings on a film, the observer's eyes were accustomed to subdued light for a period of 2 to 5 min. Variations in personal vision were within the error specified in the paper, as determined with four different operators.

The recommended film density of 1.0 to 1.2 is based on the size of the error in the estimation of the tensile strength. This is a minimum at a density of 1.0 to 1.2.

The effect of section thickness shown in Fig. 5 of the paper is based on a constant exposure factor.

The contrast index is based upon the contrast between sound and unsound regions.

Mr. H. J. Brandt.[3]—I noticed in reading Mr. Busk's paper that the films were exposed to a density of 1.2. If they were exposed to 2 or 2.5 would that affect the curves appreciably?

Mr. Busk.—The answer to that is still in the making. We are investigating that effect and with the test blocks that we used we found that we had greater differences, as far as this method was concerned, with a density of 1.2 than we did with higher densities. However, that is being checked now with porosity studies.

If the films were exposed to a density of 2.0 or 2.5, the curves would be appreciably affected. A much higher 1/v value than found at a density of 1.0 and 1.2 would correspond to any given tensile strength.

Mr. Brandt.—In correlating the tension tests, I believe you determined the index factor from the area in the negative representing that section of the test bar where fracture occurred. Did the bar actually fail in all cases in the area which showed the heaviest microporosity?

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[1] Research Physicist, Triplett & Barton, Inc., Burbank, Calif. Formerly with the National Research Council of Canada.

[2] Metallurgist, The Dow Chemical Co., Midland, Mich.

[3] Metallurgist, Aluminum Company of America, Detroit, Mich.

MR. BUSK.—Yes, providing that area were large enough. A little dot would not ordinarily cause a break.

MR. ROBERT KATZ.[4]—Could it not be that the greater contrast or greater sensitivity of the method, with films exposed to a density of 1.2, is due somewhat to the characteristics of the bulb in the viewing box and the variation of light emission of that bulb with voltage? I have found, and I think others have found, that greater density of films yields greater sensitivity in the inspection of magnesium alloy castings. However, higher densities require stronger sources of light in the viewing boxes.

MR. BUSK.—That could very easily be true. With the particular viewing screen that we used, there was no difference in the emissivity curve against voltage when changing from bulb to bulb.

MR. KATZ.—This work was done with laboratory specimens which were made under controlled conditions. Do you believe that the time is ripe to translate these data into a chart which would enable anyone to take a commercial casting and X-ray it and predict from this chart what the tensile strength of various areas on that casting would be?

MR. BUSK.—That has been done to a considerable extent in our foundry in Bay City, in which castings have been radiographed and tensile strength predicted. In a great many cases test bars were cut out and checked against the prediction, and in all cases the error was within the error specified. I neglected to mention that C alloy HTA was derived from such data of the wall of an actual commercial casting.

MR. DON M. MCCUTCHEON.[5]—I wonder whether there might not be some theoretical basis for your method found in Glocker's book on "Materialprüfung mit Roentgenstrahlen," where the factor S_0-S is used, in which case would you not have got the same results, if instead of lowering the light intensity you had examined your films with a spectrowedge or a neutral tint wedge? That might answer Mr. Brandt's question and the first part of Mr. Katz's.

MR. BUSK.—That is right, exactly the same.

MR. T. A. TRIPLETT.[6]—I am very glad to see this paper. At the annual meeting in 1939 I gave one much more crudely but with essentially the same information, and the results are comparable to those Mr. Busk has found.

One of the things I noticed particularly is that Mr. Busk is reading to a density of 1.2. We have made a considerable number of measurements on the sensitivity of films and have found a greater sensitivity at 2.5. Therefore, I think you can expect better results by working in that range.

Further, we found that this microshrinkage was definitely more serious than most people have believed from conditions other than the straight tensile strength. On his charts I noticed the author has a range of the greatest amount of porosity from approximately 19,000 psi. up to 38,000 psi., but in each case it is practically a factor of two to one.

I brought just a few of these data on more than 100,000 tests with me to show you that our investigations have covered a large field.

Among these tests on the magnesium alloys we have found one which caused a little trouble. We experimentally X-rayed some magnesium samples for one of the local field men of the Army Air Corps and one piece of this material had an enormous amount of porosity. On your index it would be from 280 to perhaps 320. Another section of the same casting had practically none, and we predicted the results of this particular material in the tension test and were fairly correct in our prediction. However, we had enough material of each condition to carry out a corrosion test, and placed the specimen in salt spray and, much to our amazement, the very good material, which radiographically showed no microshrinkage, showed absolutely the worst condition of corrosion of the surface while the material with the large amount of shrink porosity, or microshrinkage, did not appear to have corroded much.

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[4] Army Air Forces, Wright Field, Dayton, Ohio.

[5] Metallurgical Engineer, Main Laboratory, Rouge Plant, Ford Motor Co., Dearborn, Mich.

[6] X-ray Metallurgist, Triplett & Barton, Burbank, Calif.

At the conclusion of this particular test, when we pulled the specimens, the reverse was true. The material that was good on the radiograph still was relatively good, but the material that was bad on the radiograph fell apart in our hands while taking it out of the salt spray.

MR. H. H. LESTER.[7]—I should like to remark that this relationship between porosity and physical properties seems to be pretty important for the magnesium and aluminum castings, but we have not been able to find as much correlation as we should like in the case of steel. That is, we have worked on it quite a bit and we have not had satisfactory results. The results have been so erratic that we have not been able to get much consistency out of them. However, we did find in some tests that the yield strength seems to be very little affected by ordinary amounts of porosity in steel, and consistently with the author I believe we found that the tendency seemed to indicate that there was a difference in tensile strength.

We found that one of the best methods for getting the physical effect was in the bend test; large amounts of porosity definitely gave lower bend test values. In fact, we got the best correlation in any of our tests when examining with the bend test.

The matter is quite important, especially for the steel men, because the Boiler Code deals with standards based upon the amount of porosity. If the moderate amounts of porosity are not important in affecting the yield strength, perhaps those standards ought to be modified.

MR. JOHN P. MAGOS.[8]—I think Mr. Busk is to be congratulated on submitting a paper of great engineering value. We attempted a few years ago to make similar correlations on steel and we selected as one way of getting small-scale porosity by comparing the properties of gas welds to electric arc welds. We found, as Mr. Lester stated, that on the bend test you can note that the gas welds have a lesser ductility than the electric arc weld, but on actual tensile pulling there was not much to choose between the two, and radiographically the gas weld looks inferior to the electric-arc weld, but mechanically it is perfectly all right.

We then decided that possibly the differences could be determined better by some dynamic testing, and in order to get away from the small-scale fatigue test bar we undertook to construct a special apparatus which would dynamically test large structures. We built an electromagnetic vibrator and we were able to test tubing up to 4 in. in outside diameter, with about $\frac{3}{8}$-in. wall thickness. These tubes had circumferential welds made both by the gas and electric arc method and we tried to make some bad welds purposely.

Radiographically some of the welds

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[7] Principal Physicist, Watertown Arsenal, Watertown, Mass.

[8] Directing Engineer, Research Laboratories, Crane Co., Chicago, Ill.

looked pretty bad, and we were quite hopeful that we would show up some great differences. We proceeded to make the dynamic tests and in every test the failure occurred in the parent metal or in the zone of fusion and the failure was entirely attributed to the slight change in section between the weld and the tube wall. We found, therefore, that the weakest part of the structure was the mechanical design. In other words, the stress concentration in change of section, even though it is very slight, was far more weakening in the final test than all of this porosity and the other things that we note radiographically.

MR. J. D. HANAWALT.[9]—I wish to comment on Mr. Triplett's remarks about corrosion. I think that is irrelevant to the subject of porosity. I do not believe that the observation about corrosion in salt spray was related to porosity but was related to cathodic areas or impurities or something of that nature. I think that one must be sure when one is talking about the effects of porosity that other conditions are kept constant; that is, besides the question of the corrosion characteristics of the material, matters of grain size, of heat treatment or composition must all be considered in their relation to the properties which are being measured, and it is only when those conditions are known or under control that one can expect any correlation with porosity as measured.

MR. TRIPLETT.—I might point out that these materials for the test came from the same specimen, and within the possibility of casting a specimen they were of the same quality and under the same controlled conditions.

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[9] Director of Metallurgical Laboratory, The Dow Chemical Co., Midland, Mich.

A MILLION-VOLT PORTABLE RADIOGRAPHIC X-RAY UNIT

By E. E. Charlton[1] and W. F. Westendorp[1]

Synopsis

A million-volt portable X-ray unit of small dimensions and light weight is described. Its principal features are: the multisection X-ray tube, the coreless resonance transformer, and the Freon gas insulation. It permits radiography from ten to several hundred times faster than with older equipment through thick masses of steel ranging from $\frac{1}{4}$ in. to 8 in., producing a radiographic definition of from 1 to 2 per cent.

In the development of a million-volt X-ray unit for industrial radiography, portability seemed to be a particularly important objective to be attained.

The application of very penetrating X-radiation for the nondestructive examination of very thick sections of metal demands freedom of movement of the generating unit because the weight and irregular shapes of the larger castings and welded structures make it exceedingly difficult to position them easily and rapidly for the best radiographic technique.

The X-ray tube for this portable unit has been sealed off the pump, and all the component parts of the entire unit are enclosed in a grounded metal tank 3 ft. in diameter, 4 ft. in length, with a combined weight of 1500 lb.

The generating unit consists principally of a low-frequency resonance transformer with a coaxially mounted, sealed-off, multisection X-ray tube within, both contained in a steel tank and insulated with compressed gas. The X-rays are generated from a target mounted in the end of an extension chamber projecting from one end of the tank. Aside from the feature of portability, the unit being readily maneuvered into any position, it is also of a simple yet rugged design, reliable in its electrical performance, free from exposed high voltage, and has a readily accessible X-ray target.

Transformer:

The transformer (Figs. 1 and 2) has a low-voltage coil of rectangular wire (*14*) and a high-voltage coil (*9*) consisting of 125 thin flat sections spaced apart for cooling.

The resonance principle of operation may be explained as follows: The high-voltage coil has a larger inductance which is chosen to be in resonance with its terminal and distributed capacity at the operating frequency of 180 cycles per second. To build this 30 kva. resonant system up to the large amplitude of oscillation of 1,000,000 v. requires the relatively small input power of $3\frac{1}{2}$ kw. which equals the losses in the transformer plus the power for the X-ray tube and can therefore be supplied by the small primary winding placed at the grounded end of the high-voltage winding. The

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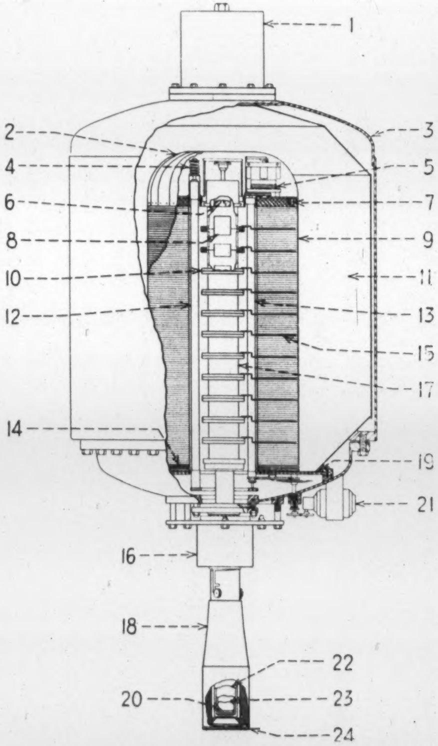
[1] Research Laboratory, General Electric Co., Schenectady, N. Y.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1. Cooler.
2. Slotted brass shield.
3. Steel tank.
4. Spring for tie rod.
5. Variable reactor.
6. Cathode assembly.
7. End turn filament coil.
8. First intermediate electrode.
9. Secondary coils.
10. Shields around the X-ray tube.
11. Laminated shield.
12. Glass tie rod.
13. Insulating filament-control shaft.
14. Primary winding.
15. Tap lead.
16. Focusing coil.
17. Glass envelope.
18. Lead shield.
19. Laminated steel bottom.
20. Tungsten target.
21. Filament control motor.
22. Water jacket.
23. Extension chamber.
24. Lead diaphragm.

FIG. 1.—The Elements of the Million Volt Portable X-ray Unit.

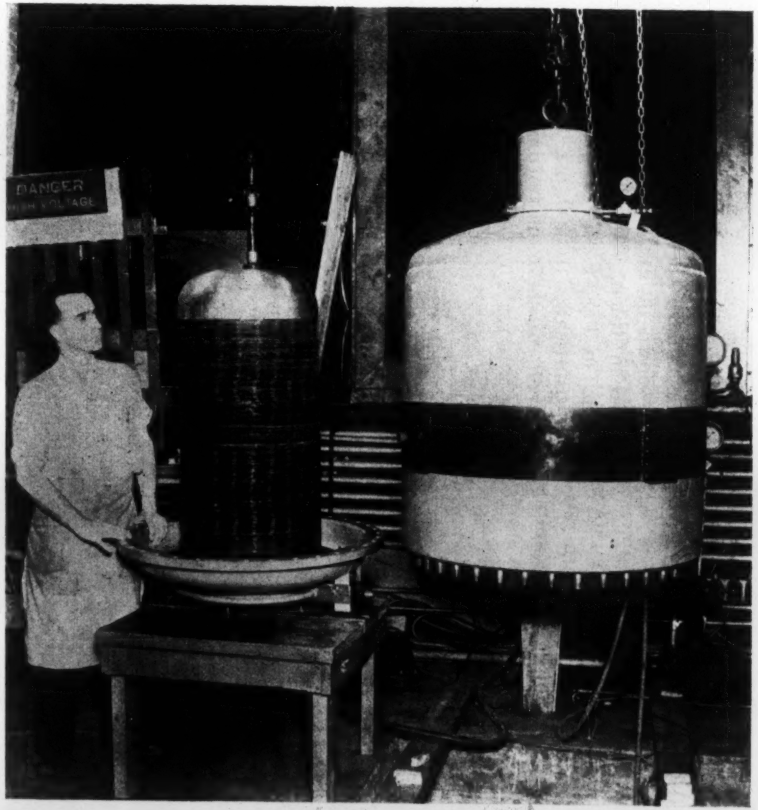
DANGER
HIGH VOLTAGE

FIG. 2.—The Resonance Transformer.

mechanical analogy of the resonant transformer is the swinging pendulum, or a system in which a mass is oscillating at resonance with a spring support. In such mechanical systems a small force of the proper frequency can, with relatively little power, maintain large amplitude oscillations containing much stored energy.

The inductance required for resonance can be obtained in a coil of this size without the use of an iron core, and the central space of the high-voltage coil is therefore available for the X-ray tube, thus facilitating the making of connections to various tube electrodes and providing the benefit of electrostatic shielding by the transformer coil.

The lower end of the high-voltage coil is grounded; the upper or high-voltage end is shielded by a rounded and radially slotted brass spinning.

The 180-cycle power is derived from the 60-cycle line by means of a synchronous motor generator set which also renders operation independent of line-voltage fluctuations.

To avoid the power loss which would ensue were the magnetic flux of the transformer to penetrate the surrounding steel tank, the latter is provided with an inner lining (*11*) consisting of narrow overlapping silicon steel strips spot-welded to a thin shell and at the bottom with a ring of similar radial strips (*19*).

The multisection high-voltage coil serves a quadruple purpose: to generate voltage, to grade the potential so as to prevent creepage, to shield the X-ray tube electrostatically from the grounded tank, and to support mechanically the rounded top shield. The coil is held under strong compression by seven spring-loaded tie rods, which make it possible for the outfit to be operated in any position as shown in Figs. 8 and 9.

Steel Tank:

The tank of the million-volt portable unit shown in Fig. 3 is made of sheet steel $\frac{1}{4}$ in. thick, 36 in. in diameter, and 48 in. in height and has been tested hydrostatically to 200 psi. The joint between the shell and bottom flanges is made gastight by means of a rubber gasket.

Gas Insulation:

The X-ray tube and resonance transformer are insulated from the grounded steel tank with gas insulation. The organic chemical dichloro-difluoro methane known commercially as "Freon-12" or "F-12" is introduced into the tank to a pressure of 60 psi. after the unit has first been exhausted of air to a 28-in. vacuum. The Freon-12 gas at this pressure provides adequate insulation to prevent sparkover in the tank at voltages far in excess of the rated million volts. A pressure-stat on the main tank blocks operation of the transformer if the pressure drops either by leakage or due to abnormally low temperature below 50 psi. The gas F-12 is odorless and nontoxic; however, it must never come in contact with an open flame, the resulting decomposition products being poisonous.

X-ray Tube:

A twelve-section X-ray tube (Figs. 1 and 4) sealed off the pump for continuous operation at 1,000,000 v. and 3 ma. was especially designed to go with this resonance transformer. It has an electron-emitting cathode, a copper-backed tungsten target mounted in the lower end of an extension chamber, and accelerating cylindrical electrodes in each of the intermediate sections.

The tube envelope consists of twelve sections of molded borosilicate glass tubing joined to fernico rings which

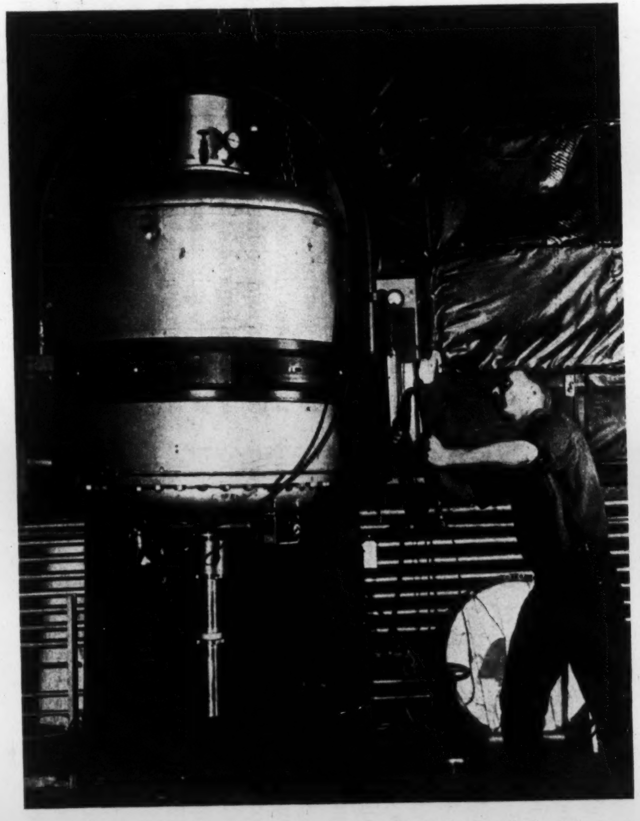

FIG. 3.—The Completely Assembled Unit in Steel Tank.

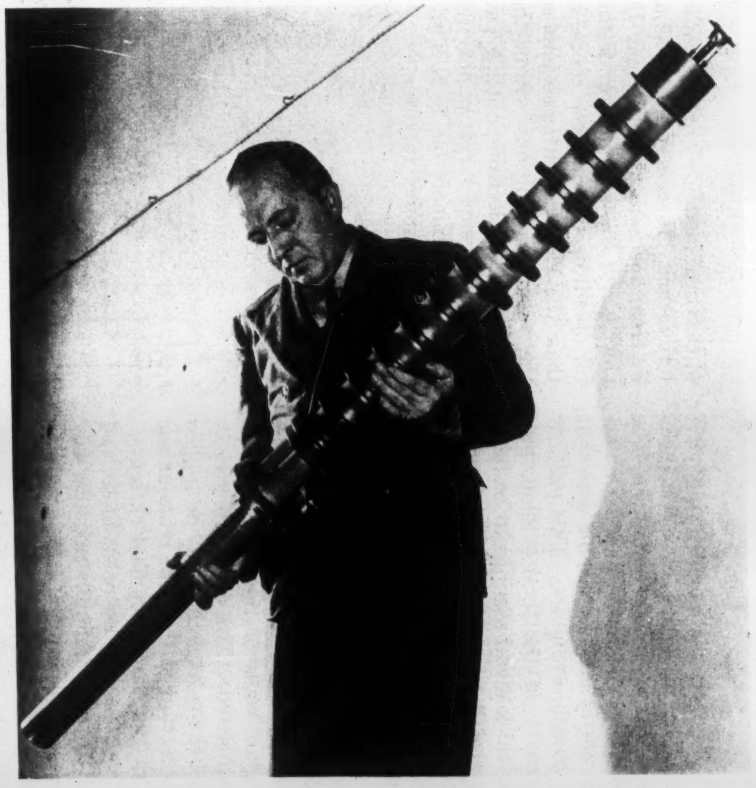

FIG. 4.—The Sealed-Off X-ray Tube of Million Volt Portable Unit.

carry the intermediate accelerating electrodes. The insulating envelope is 30 in. long and $3\frac{1}{2}$ in. in diameter. The fernico is a special alloy which has an expansion coefficient comparable with that of the borosilicate glass. This particular property of the fernico alloy, permitting thick metal sections to be fused directly to the glass, has been of major importance in the design of this million-volt X-ray tube. It has allowed the tube to be made much smaller than might otherwise be possible mechanically and has also permitted a rigorous exhaust of the tube to be carried out before it is sealed off the pump. The inside glass walls of the tube envelope are sandblasted. This procedure greatly increases the voltage that may be applied to each section of the tube without the production of dangerous field currents.

The cathode spiral consists of six and one-half turns of 8.5-mil tungsten wire mounted in an electrostatic focusing cup. The eleven intermediate accelerating electrodes are made of stainless steel. The target of copper-backed tungsten is mounted in the lower end of the extension chamber, and both target and chamber walls are water cooled. The tube is supported by a metal flange bolted to the bottom of the grounded metal tank. This flange has been soldered to the copper extension chamber which projects through it.

The tube is sealed off the pump after a specially developed evacuation process has been carried out, during which the glass envelope and metal parts are thoroughly outgassed, and all sections are aged at voltages 100 per cent higher than the operating voltage. This process insures stability of operation and long life. While it is too early to predict the useful operating life of these sealed-off tubes, we can say that some of them have now been in daily radiographic service at 1,000,000 v. for over a year without showing the slightest signs of electrical instability or mechanical deterioration. It seems quite possible that external mechanical abuse may be the chief factor that will determine the useful operating life of these tubes, rather than the electrical stresses imposed or the vacuum conditions within the envelope. The electron-emitting cathode and the tungsten target appear now to play a very minor role in this tube as factors determining tube life.

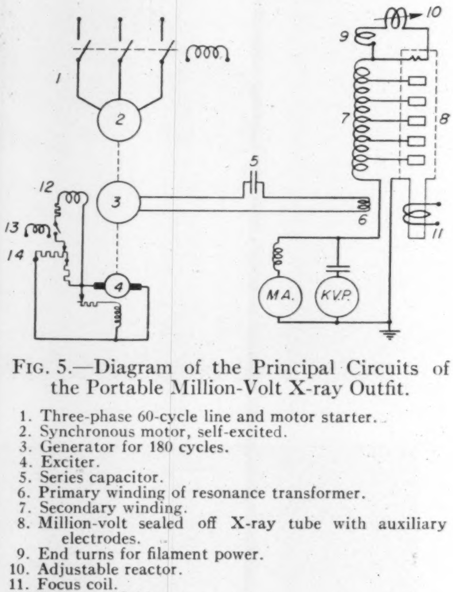

FIG. 5.—Diagram of the Principal Circuits of the Portable Million-Volt X-ray Outfit.

1. Three-phase 60-cycle line and motor starter.
2. Synchronous motor, self-excited.
3. Generator for 180 cycles.
4. Exciter.
5. Series capacitor.
6. Primary winding of resonance transformer.
7. Secondary winding.
8. Million-volt sealed off X-ray tube with auxiliary electrodes.
9. End turns for filament power.
10. Adjustable reactor.
11. Focus coil.
12. Generator field.
13. Starting relay.
14. Kilovoltage adjusting potentiometer.

Electrical Circuit:

Figure 5 shows a schematic diagram of the principal circuit elements. Pushbutton controls, selector switches, control motor drives, interlocks, protective devices, and indicator lights have been omitted for the sake of readability.

The motor starter (*1*) connects the 3-phase, 60-cycle power line to the motor generator set. The self-excited synchronous motor (*2*) drives the 180-cycle generator (*3*) and the exciter (*4*). From the generator the 180-cycle power passes through a series capacitor (*5*), through a long, flexible multiple-conductor cable to the primary of the X-ray outfit (*6*). The series capacitor serves to neutralize the generator reactance and the primary leakage reactance of the transformer, thus loading the generator under all operating conditions with a unity power factor load and making the operation independent of the small variations in frequency which occur on power systems. The high-voltage winding (*7*) is connected through the metering circuit to the grounded extension chamber of the tube (*8*), through the taps to the intermediate electrodes of the tube and at its upper terminal to the cathode. The filament is operated off end turns (*9*) on top of the main coil, and the filament current adjusted by means of a variable reactor (*10*) driven through a glass insulating shaft by an external filament-control motor.

The metering circuit consists of a d-c. milliameter with a reactor in series and an a-c. milliameter with a capacitor in series. The X-ray tube electron current is a unidirectional current and registers on the d-c. milliameter; the secondary charging current (55 ma. rms. at 1000 kv.p.) passes through the capacitor and the a-c. milliameter, which is calibrated in kilovolts peak.

The field (*12*) of the generator is connected through a push-button operated "X-ray-On" relay (*13*) to a motor-driven potentiometer (*14*) which is connected across the exciter. The exciter can also be used to furnish the direct current for the focus coil (*11*). The motor which moves the contact on the potentiometer (*14*) is operated from the control panel by means of a small "kv.p. Control" lever. Interlocking contacts are provided so that the unit cannot be started unless the potentiometer contact is in its lowest position, corresponding to approximately 500 kv. Another control lever is provided on the panel to operate the filament-control motor for the adjustment of the current through the X-ray tube.

Interlocks are provided for the X-ray room doors, the cooler motor, the Freon pressure, and the cooling water. There is also relay protection against overvoltage and overcurrent beyond the range selected. A selector switch is provided on the control panel to choose a 0–20 microammeter, a 0–100 microammeter, a 0–1 milliameter, or a 0–5 milliammeter to read the X-ray tube current. The same switch adjusts simultaneously the direct current through the magnetic focus coil (*11* in Fig. 5) surrounding the extension chamber which controls the diameter of the electron beam, thus automatically setting the focal spot size on the target for each of the current ranges. The selector also switches various shunts on a tube-current-operated relay which protects the outfit and the meters against operation beyond the selected range.

Two timers of 0–5 and 0–30 min. ranges and a selector switch are provided; the selected timer breaks the interlock circuit and opens relay (*13*) after the preadjusted time interval has elapsed.

OPERATION OF THE UNIT

A typical operating sequence of the million-volt portable outfit is instructive. Assume that the operator plans to run his next exposure for 1 min. at the full rating of 3.0 ma. and 1000 kv.p. Before he places the film, he makes adjustments as follows: He calls all people out of the X-ray room by means of a horn signal, closes the doors, selects the 5-ma. meter on the panel, starts the motor generator set, pushes the "X-ray On" button, lets the kv.p. meter come up to the starting voltage of 500 kv.p., and then raises the kv.p. handle; after he has reached 1000 he adjusts the filament control till he has 3.0 ma. He is then ready for the exposure of the film, pushes the "Off" button, and goes into the X-ray room to place his films. For the actual exposure he repeats the foregoing procedure without further filament adjustment except that he selects the 5-min. timer which he has adjusted for the desired 1-min. exposure. After each run the kv.p. control motor returns the potentiometer of the generator field automatically to the starting position.

DESIGN FEATURES

The Resonance Transformer:

In a high-voltage X-ray unit, the resonance transformer offers several advantages over the conventional type of transformer.

The absence of the iron core, permitting the tube to be placed in the axis, conserves the space otherwise required for insulation between core and high-voltage winding and for the insulation of the tube. The tank diameter of the resonance transformer is approximately 62 per cent of that of the conventional transformer and the volume about 44 per cent or even less if the space required by the tube is considered.

The wave form in the high-voltage circuit is always sinusoidal regardless of the input voltage. Furthermore, the oscillating current in the high-voltage winding is so large (55 ma. at 1000 kv.p.) that the rated half-wave full load current of 3 ma. average does not produce any measureable difference between useful and inverse voltages.

In this resonance transformer, which has 30 oscillating kva. and with an X-ray load including losses of approximately 3.5 kw. maximum, the voltage takes a few cycles for build-up, even if the equipment is switched on suddenly. Therefore switching surges or other disturbances do not raise the output voltage more than a fraction of 1 per cent, whereas in ordinary transformers 50 per cent or more voltage surges may occur frequently in case improper switching takes place.

Solid Insulation:

The subdivision of the high-voltage winding into 125 thin coils results in a very uniform voltage gradient along the length of the stack. This high degree of subdivision of the solid dielectric throughout the secondary of the transformer greatly reduces the probability of surface electrical creepage occurring and is perhaps the most important single factor in the design which permits such a great reduction in its over-all size.

The stack of transformer coils is securely bolted together with seven glass tie rods. These rods are made of hard glass free of air lines and blow holes and with their surface sandblasted to further increase their resistance to surface creepage discharges. The voltage distribution throughout the length of the rods is uniformly graded by placing them close to the transformer coil.

The insulating shaft for adjusting the filament current is also made of hard glass and has dimensions and treatment similar to those of the transformer tie rods. This control shaft, mounted inside the transformer stack and driven by a reversible motor, provides remote control for the variable inductance in the filament circuit to give the desired milliamperage.

Gas Insulation:

The use of gas in place of transil oil for insulation has been the principal factor in the large reduction in weight. Freon-12 has a dielectric strength 2.5 times that of nitrogen for the same pressure. For an operating voltage of 1000 kv.p., 50 psi. gage pressure affords an ample factor of safety. This calls for a weight of F-12 of about 40 lb. More than 10,000 lb. of transil oil would be required to give the same insulation in a necessarily larger tank.

Freon-12 decomposes under the action of corona, setting free chlorine and fluorine. Experience with units of this type over a period of years employing F-12 as an insulating medium has demonstrated that there is not enough such decomposition taking place under the conditions pertaining in this outfit to cause troublesome deterioration of any of the insulation or other materials present.

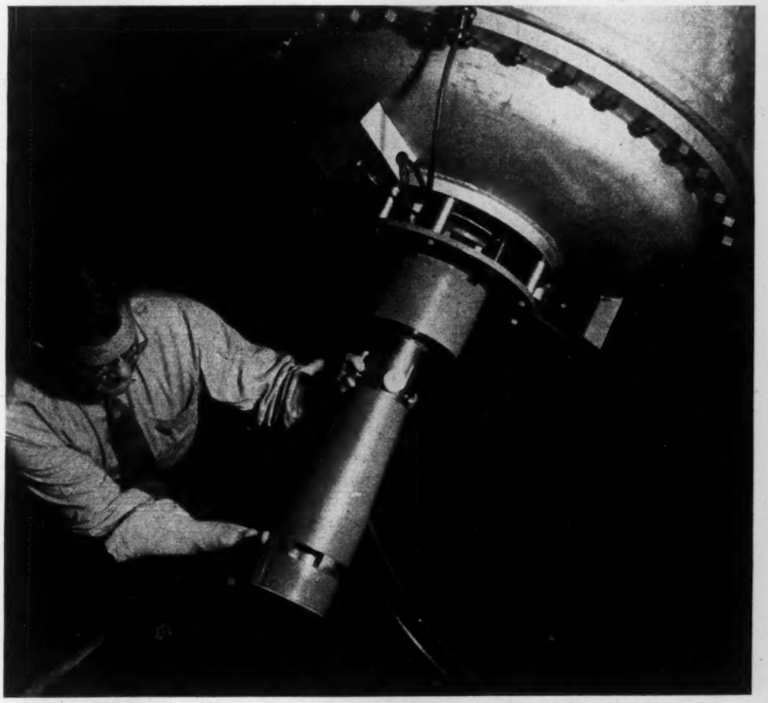

FIG. 6.—Lead Shield Around Extension Chamber to Improve Quality of Radiograph.

X-ray Tube:

Since the tube is mounted in the axis of the resonance transformer, it is located in a uniform electric field and a weak magnetic field parallel to the tube axis. This magnetic flux does not interfere with electron focusing.

Toroidal shields surrounding and connected to the various electrodes electrostatically shield the electron beam. The size of the electron beam is determined largely by cathode geometry and by the ratio of the voltage used in the first to that in the remaining sections. With an extension chamber 24 in. long and without magnetic focusing, the focal spot is about $\frac{7}{8}$ in. in diameter. It can be changed to any desired size by use of the magnetic focusing coil placed around the chamber. For industrial radiography the size of the focal spot is carefully controlled and its dimensions are reduced as far as possible without overloading the tungsten target and shortening the useful life of the tube. Radiographic techniques with million-volt X-rays require the X-ray tube to be operated over a range of current from a few microamperes up to 3.0 ma. Accordingly, several sizes of focal spots are provided, ranging from about $\frac{1}{8}$ in. up to $\frac{1}{2}$ in. in diameter, depending on the current flowing through the tube.

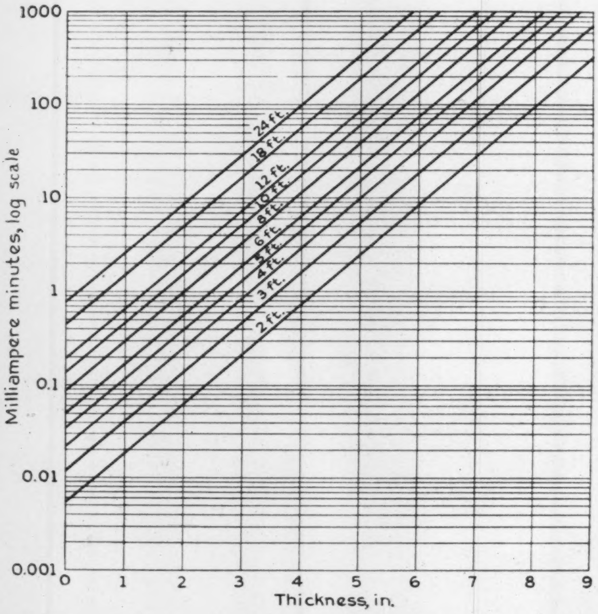

FIG. 7.—Radiographic Exposure Chart for Cast Steel Using Million Volt Portable X-ray Unit.

Zero degree beam—1000 kv.p. 0.005-in. lead filter screens. Industrial X-ray films. Film density of 1.0. Family of curves for different target-film distances.

X-RAY PROTECTION

The X-ray protection for this unit when used as a radiographic tool must be supplied largely by building the enclosure in which it is used with thick walls of concrete or lead. This becomes necessary because diversified radiographic techniques with metal structures of different shapes demand that the extension chamber be not covered with a large and heavy protective shield.

RADIOGRAPHIC APPLICATION

A lead shield (Fig. 6) with a 1-in. wall thickness and weighing 40 lb. is placed around the extension chamber. This does provide some primary protection, but its principal purpose is to improve the quality of the radiograph that may be obtained by reducing the effects of scattered and stray X-radiation. This lead shield is so designed that it may be quickly adjusted to change the diameter of the cone of radiation of either the reflected or the transmitted beam of X-rays that is being used for a given radiographic procedure. It is always desirable to cone down the useful beam of radiation to a diameter that will just cover the portion of the material that is being radiographed. When this cone is used in combination with a lead shield placed beyond the film back of the cast-

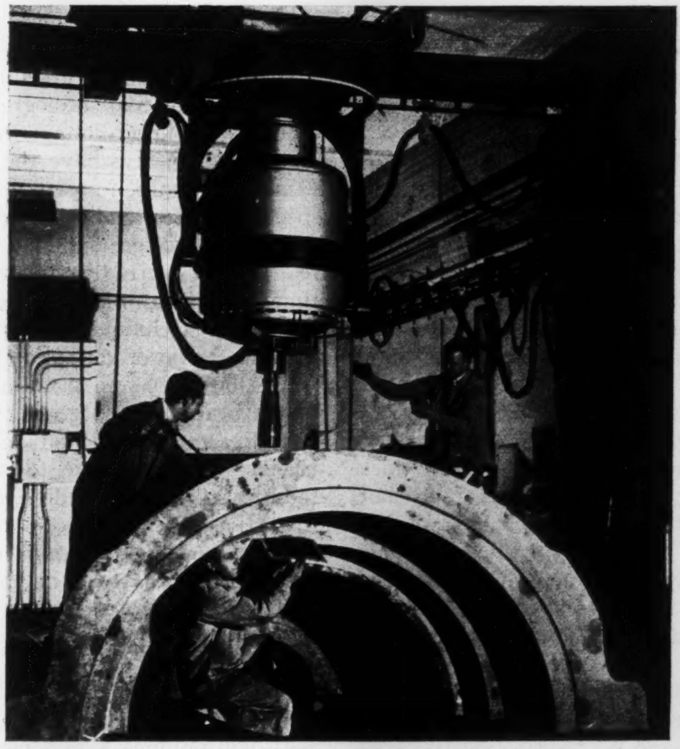

FIG. 8.—Metal Radiography with the Million Volt Transmitted Beam—Vertical Position.

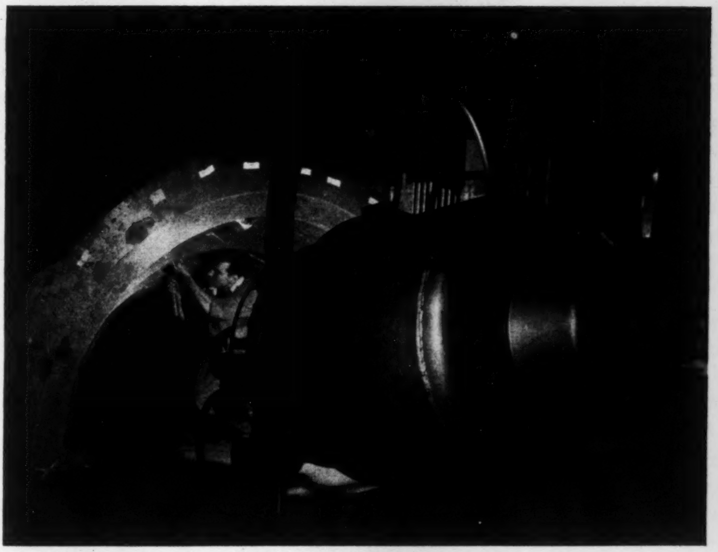

FIG. 9.—Metal Radiography with Million Volt Reflected Beam—Horizontal Position.

ing which also encircles the X-ray beam, the undesired effects of stray radiation on the radiographic shadows are eliminated.

When radiographing the thinner sections of steel with million-volt X-rays, the use of a lead filter placed between the object and the film is particularly desirable to reduce the effect of secondary radiation through the body of the steel itself.

This X-ray unit, rated to operate continuously at 1,000,000 v. and a maximum current of 3.0 ma., produces very penetrating X-rays of high intensity. Radiographs of thick sections of steel, ranging from 1 to 8 in. in wall thickness, may be obtained with it in short exposure times. Figure 7 illustrates the exposure times required with million-volt X-rays to take a radiograph through different thicknesses of cast steel at varying target-film distances.

Typical action scenes illustrating the radiographic application of the unit in an industrial X-ray laboratory are shown in Figs. 8 and 9.

Stereoscopic radiographs of metal sections of widely varying wall thickness may be obtained with good definition. This is made possible and practical because of the large amount of penetrating X-radiation that is generated in this unit.

It has been found that radiographs taken with million-volt X-rays will clearly define defects ranging from 2 per cent down to less than 1 per cent in steel structures having walls varying in thicknesses from $\frac{1}{4}$ in. to 8 in. In contrast to industrial radiographic techniques with lower-voltage X-rays, the million-volt X-rays have the added advantage not only of greater radiographic speed but also of allowing greater latitude in the range of metal thicknesses readable on one radiograph without the time-consuming complication of a "blocking" technique as generally practiced in the lower-voltage range.

The intensity and penetrating power of the beam permits greater target-film distances to be used and still keep the time of exposure down to seconds and minutes. This markedly reduces the distortion of the shadows recorded on the film and also increases the area of the material that can be radiographed with one-exposure technique.

CONCLUSION

This portable million-volt X-ray unit, operating continuously at 3.0 ma. current, will produce X-rays which both in quantity and quality are equivalent to those obtained from large air-insulated and immovable X-ray units previously designed for medical applications. The ability to move this unit around easily in all directions makes it a flexible and powerful tool for the rapid radiographic examination of castings and metal structures varying greatly in size and in wall thickness.

DISCUSSION

MR. H. H. LESTER.[1]—What is the smallest focal spot size that you can get with this million-volt outfit?

MR. W. F. WESTENDORP.[2]—We can get $\frac{1}{8}$ in. In the laboratory we can get a spot that, with our method of determination, looks like a 2-mm. spot. Since our method of determining the focal spot does not tell us exactly whether it is 1 or 2 mm., it might be even smaller than 2 mm. I have been asked how we determined the focal spot size of a tube like this with a million-volt very penetrating radiation. You are all probably familiar with the focal spot camera. It is just the ordinary camera obscura applied to X-rays. This will not work for million-volt X-rays because the pinhole would not be a pinhole any more. You could not make a suitable pinhole in a 1-in. lead plate. You would not be able to get radiation to go at angles through a 1-in. lead plate. You would have to drill two cone-shaped holes and then, of course, you would not get the effect of a small hole. The effect would be of a hole of much larger diameter because of the penetrating radiation. It would not go through just the little hole; it would also go through a considerable amount of lead, and therefore it is impossible to use a pinhole camera for million-volt X-rays. We had to develop a new technique for determining the size of the focal spot.

We made a block of lead, a 4-in. cube, and had 10,000 vertical holes built in. We did not drill those. We took a large number of lead sheets and made a steel die with 30 triangular grooves per inch, and we pressed the steel die in the lead sheets. The sheets were 0.030 in. thick by 4 by 4 in., and by pressing the grooves in the lead sheets and then putting the lead sheets together we got the effect of 10,000 small vertical holes, all perfectly parallel in this lead block.

If you place that kind of diaphragm close up against the target and with film underneath it, you will get a parallel projection of the focal spot on the film. Only those X-rays that go right straight through the holes will affect the X-ray film. All the others will get blocked off and, depending on the size of the holes and the number of holes per square inch, you get a certain definition, and that is what I was referring to when I said our method did not allow us to get more definition than the diameter of the hole, which is about 0.030 in.

MR. LESTER.—Is there any possibility that a pinhole can be made smaller in the future?

MR. WESTENDORP.—Yes. It just means a lot more work, thinner sheets of lead, and a finer die. It is a lot of work to make a tool like that because the lead sticks to the die and has to be peeled off very carefully and has to be straightened again. If you straighten it too hard you ruin it. It takes a skilled mechanic about a week to make this grid, and we do not like to go through it if there is not a very great interest in the exact diameter of the focal spot. We have had no burnt-out targets with this tube, so it is possible that we are a little bit conservative. We would rather be conservative than burn out the targets.

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[1] Principal Physicist, Watertown Arsenal, Watertown, Mass.

[2] Research Laboratory, General Electric Co., Schenectady, N. Y.

MR. C. A. ADAMS.[3]—Is the tube its own rectifier?

MR. WESTENDORP.—Yes. You see, the multisection principle makes it possible to apply an inverse voltage of 1,000,000 v. as well as a forward voltage of 1,000,000 v. The voltage is divided over twelve sections, and each section does not get over 80 or 90 kv. In the construction of the tube we actually apply about 180 kv. to each section at the time.

MR. ADAMS.—Is the potential gradient pretty uniform up and down the tube?

MR. WESTENDORP.—Yes, because of the large number of coils.

MR. ADAMS.—The actual current is an electrostatic induction current, not electromagnetic?

MR. WESTENDORP.—The charging current through the coil becomes a dielectric current when it goes to the inside tank wall.

MR. DON M. MCCUTCHEON.[4]—Does the measured focal spot image depend upon the intensity of the beam to such an extent that difficulty would be experienced in actually assigning a true value for the focal spot size?

MR. WESTENDORP.—No. We have made measurements of that. We have used different exposure times, and if you expose long enough, then of course the whole film will blacken because of the radiation that gets through the lead. But there is a length of time, from 10-sec. to 1 or 2-min. exposure, that will produce consistent spots of a definite diameter.

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[3] Professor of Engineering, Emeritus, Harvard University, Cambridge, Mass.; and Consulting Engineer, Edward G. Budd Manufacturing Co., Philadelphia, Pa.

[4] Metallurgical Engineer, Main Laboratory, Rouge Plant, Ford Motor Co., Dearborn, Mich.

HIGH-VOLTAGE X-RAYS IN THE BOILER SHOP

By O. R. Carpenter[1]

Synopsis

The development of radiographic equipment for industrial use from the 200-kv. machine of 10 yr. ago, similar in many respects to the medical equipment of that day, to the present million-volt industrial X-ray machine has kept pace with the increasing demands for radiographic examination of heavy wall fusion welded pressure vessels with plate thicknesses up to 5½ in. Application of higher-voltage X-ray equipment to industrial radiography introduced the problem of blocking excessive secondary radiation from the film. This requirement was met by the development of the Bucky grid and similar devices. Although a considerable increase in exposure time resulted from the use of these grids, X-ray films of good detail were obtained with 300-kv. and 400-kv. equipment.

The advent of million-volt X-ray equipment with the emission of extremely short wave length X-rays necessitated a radical change in our X-ray technique. A special fine-grained no-screen film (direct-exposure film) was developed for this purpose by the Eastman Kodak Co., representing a significant advancement in the solution of radiographic problems. The use of lead foil screens in conjunction with this film obviates the necessity for Bucky grids and calcium-tungstate screens and permits the standardization of X-ray technique, thus assuring consistently satisfactory results.

In June, 1941, we were installing in high anticipation the million-volt X-ray equipment now in daily use at the Barberton Works of the Babcock & Wilcox Co. The purpose of the installation of this machine was to permit a solution of the radiographic problems experienced prior to this time in the examination of fusion welds of the main longitudinal and circumferential seams of heavy-walled pressure vessels. These welds are of thicknesses up to 5½ in. A typical boiler drum having 5 in. thick welds is shown in Fig. 1. This drum is made up by means of three longitudinal seams and four circumferential seams. One hundred and twenty exposures are required for a complete radiographic examination of this drum, each exposure covering a 12-in. length of weld.

Until the million-volt X-ray equipment became available for industrial purposes, the highest voltage equipment in general industrial use was 400 kv. With this machine approximately fifteen days were needed for the complete radiographic examination of this drum. Some time savings could be made if gamma rays were substituted for X-rays for the circumferential seams, the difference being from two days up, depending upon the size capsule of radium available. Under present conditions and the consequent need for an increase in production of such pressure vessels as well as the application of fusion welding to heavier and heavier sections, the above-described radiographic technique was intolerable. Hence the million-volt X-ray installation has answered a real production problem. With it, fusion welds four or more inches thick are X-rayed in a matter of minutes, not hours as formerly required by 400-kv. X-rays.

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[1] Engineer, The Babcock & Wilcox Co., Barberton, Ohio.

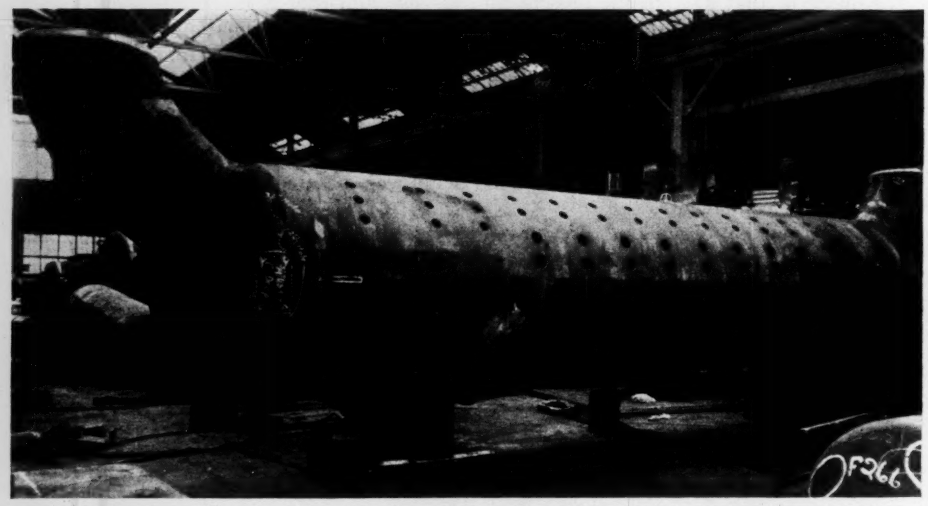

FIG. 1.—Eighty-Ton Boiler Drum Having 5-in. Thick Longitudinal and Circumferential Welds.

Not many years ago, however, the installation of a lesser voltage machine in this plant was thought of, in much the same manner, as an answer to the production problems of radiographic examination of heavy drums of that day. In 1932 a 4¼ in. thick mercury boiler drum required radiographic examination. Then the only industrial equipment available was a 200-kv. machine similar in many respects to medical equipment of that day. With this equipment 3-in. thick welds were sometimes radiographed, if the tube voltage was increased somewhat over its rating, in about 1 hr. Needless to say, the results obtained would not be considered good today and the construction of a 4¼-in. thick welded drum was out of the question radiographically. The General Electric Co., in response to the need of a higher voltage machine, built and installed the first industrial 300-kv. machine. It was regarded as a considerable advance in equipment design; it had a large air-insulated power plant capable of operating at 500 kv. A new tube was developed which could be operated at 300 kv. With it, the 4¼-in. mercury boiler drum welds were radiographed at a 30-in. film distance in 20 min. Such an exposure time is remarkably fast, even with today's equipment under 1,000,000 v., if it is only film blackening which is desired. Two per cent penetrameters could be seen on the films obtained, but these images, considered in comparison with today's possibilities, did not truly measure the film quality. These gages, while 2 per cent of the plate thickness in thickness, had holes or slots of an arbitrary size, usually $\frac{3}{16}$ or $\frac{1}{8}$ in., respectively. While images could be seen on the film, much of its density was the result of secondary radiation exposure and a very small percentage due to direct radiation exposure. Consequently, images of large defects or penetrameters could be seen but these lacked fine detail.

During the years following this installation and until the impetus of the present emergency and the installation of million-volt equipment, nothing spectacular was accomplished to lessen this exposure time. It is true that more compact and serviceable equipment was developed; typical is the 400-kv.p. machine shown in Fig. 2. This machine had the power plant and tube immersed in a tank of oil. In place of the 300-kv. tube, a 400-kv. tube was made available with a somewhat smaller focal spot. With this machine a $4\frac{1}{4}$-in. weld could be radiographed in about 15 min. exposure when using a technique similar to that used with the original 300-kv. equipment. However, users of this equipment were soon to realize that improved technique was needed for better film quality. The consequent results were gradual increases in exposure times, until a year ago this time had increased to approximately 3 hr. for a 4 in. thick weld when using 400-kv.p. X-rays.

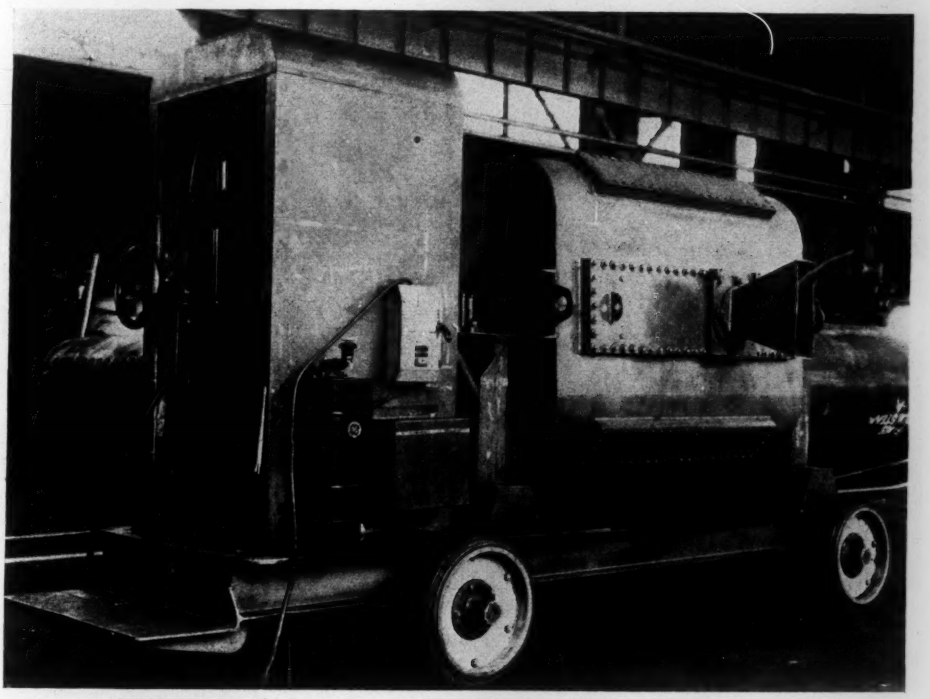

FIG. 2.—400-kv.p. Oil-Immersed X-ray Machine for Industrial Purposes.

The increase in exposure was the result of the employment of means and methods for the blocking of secondary radiation from the film. Any such device proportionately reduces film blackening, and therefore required longer exposures to bring the film to a readable density. Various methods may be used to block secondary radiation from the film, the most satisfactory being the Bucky grid. The more effective these devices, the longer the exposure time, and the greater the film detail obtainable. Nevertheless, it is of interest to point out that the sacrifices in exposure time made necessary by commercial grids did not altogether give the desired film detail. This may be illustrated by a comparison of Fig. 3 and 4. In the former there is shown a radiograph of a 1-in. thick plate in which there is a weld with a prepared group of defects. This same plate has been radiographed through 4 in. of steel using the Bucky grid technique and is shown in Fig. 4. The obvious loss in detail of the defects in the weld in the latter radiograph is readily apparent, even though the technique used was the best known until recently for this type work. Among the many factors contributing to this loss of detail we may list:

FIG. 3.—Radiograph of Prepared 1-in. Thick Test Plate.

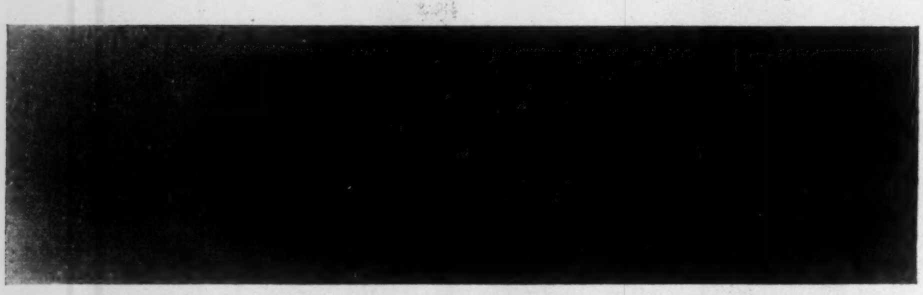

FIG. 4.—Radiograph of Plate Shown in Fig. 3, but Through a Total of 4 in. of Steel

Note loss of detail. Two per cent and four per cent deep holes of different diameters drilled on either side of defect pattern.

1. The inherent absorption characteristics of the X-ray beam in a 1-in. plate as compared to a 4-in. plate.
2. The vast difference in the secondary radiation problems of the thinner plate and lower voltage X-rays as compared to the thicker plate and 400-kv. X-rays.
3. The destruction of detail as caused by the image of the Bucky grid.
4. The ability of the film to record the detail desired, that is, its characteristics as to grain size and sensitivity to low-voltage secondary X-rays.

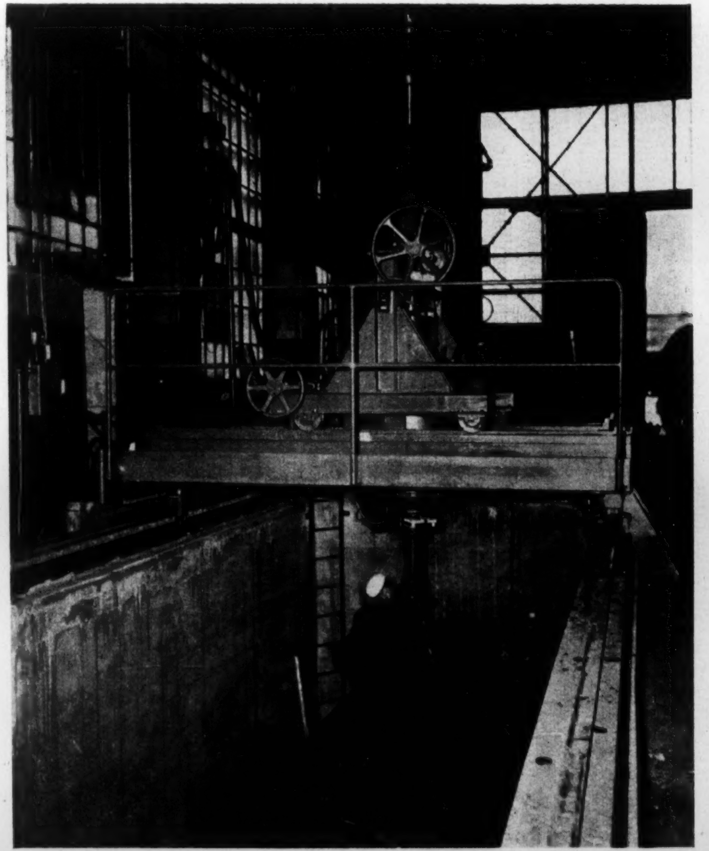

FIG. 5.—View Looking into Radiation Pit and Showing 1000 kv.p. Machine in Position over Boiler Drum.

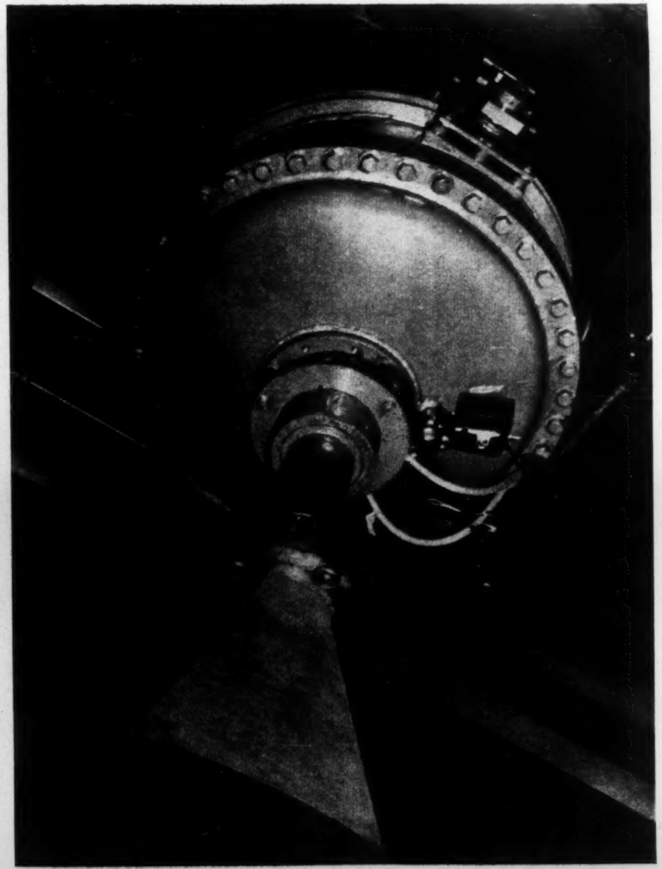

FIG. 6.—View of Transformer Head of 1000 kv.p. X-ray Machine.

It should be realized at the outset of any discussion of X-ray sensitivity that the X-ray beam itself has certain absorption characteristics in the steel which no mechanical device or other methods can alter, other conditions remaining constant. In other words, we have a tool with certain limitations and one should not expect results that exceed these limits. Radiography of welds is primarily a device useful for the disclosure of significant defects and one should not expect fineness of detail to be apparent in the radiographs of fusion welds which require a magnifying glass to make them visible. We may say, therefore, that part of the loss of detail displayed in the above illustrations is due to the increase in thickness of the materials combined with constant or decreasing X-ray absorption differentials of the beam of X-rays.

Aside from the X-ray beam itself, however, there are certain elements in the technique having considerable influence on film sensitivity. One of these is the efficiency of the Bucky grid. At its best, the mechanics of the grid are such that only part of the secondary radiation is blocked from the film. An instrument of this type which would fully block all secondary radiation would need to have such fine grid lines as to be nearly infinite in their extent. Of course, as the number of lines of the grid increase so would the amount of primary radiation reaching the film decrease, causing a corresponding increase in exposure time. Again, since grids used today have lines in but one direction, some secondary reaches the film from the unblocked direction. An ideal grid would consist of an infinite series of lead cones of very small sections, but such an instrument would have a very complicated alignment and oscillation over the film. It would increase the exposure time for welds in heavy plates far beyond any practical limits. No other similar mechanical device, equal to or better than the present commercial grid, has as yet been devised for the purpose of the elimination of secondary radiation from the film.

The film itself, its ability to record the type of radiations reaching it, and the method used for its exposure, has much to do with the loss of detail of the 4-in. film over the 1-in. film. These exposures have in both cases been made with calcium tungstate intensifying screens and films sensitive to the blue light derived therefrom. Due to the graininess of the screen and that of the film itself some detail is lost, even at lower-voltage radiation. When the wave length of the X-ray beam shortens, that is, as the X-ray potential increases, this effect is amplified causing a corresponding loss of fine detail. Aside from the film itself and the X-ray voltage involved, other factors of technique such as target-to-film distance, secondary radiation, and focal spot size contribute to the film unsharpness found in the 4-in. plate as compared to the 1-in. plate.

The above-described problems in loss of film detail of radiographs of heavy plate were largely with us until the installation of the million-volt equipment. (See Figs. 5 and 6.) The experience gained from the use of this equipment has given us an entirely new point of view of X-ray technique and exposure of heavy plate welds and brings us to the point of this discussion.

It was assumed at the outset of our experience with this machine that, because of the extreme short wave length, our accustomed X-ray technique would need to be discarded and in its place something on the order of that used for gamma-ray exposures substituted. For heavy plate thicknesses this technique then consisted of double industrial type no-screen (direct-exposure) film and lead foil screens. While good gamma-ray exposures were obtained, such technique did not give much improvement in results over 400-kv.p. X-rays with the grid technique as far as detail was concerned, and gave poorer results as far as film contrast was concerned. The film itself, because of its type, was very grainy and accounts largely for the contrast loss when used with the short wave length gamma rays. It was thought, however, that, since million-volt X-rays were, on the average, longer in wave length than gamma rays, this technique might be satisfactory. When tried it was found to give exposures not greatly different in appearance from gamma rays, they being very grainy with correspondingly poor detail. It was realized that since million-volt X-rays would penetrate heavy welded drums in minutes of exposure rather than hours, a slower fine-grained film could be accommodated without much loss in production time. The Eastman Kodak Co. then was able to furnish us such a film for trial. This film has an exposure factor of about 1.2 as compared to 0.7 for the original industrial no-screen type film. The first of the trial exposures made is shown in Fig. 7. The immediate gain in detail is readily apparent and we believe the combination of million-volt X-rays on the proper film solves many of the radiographic problems of heavy plates.

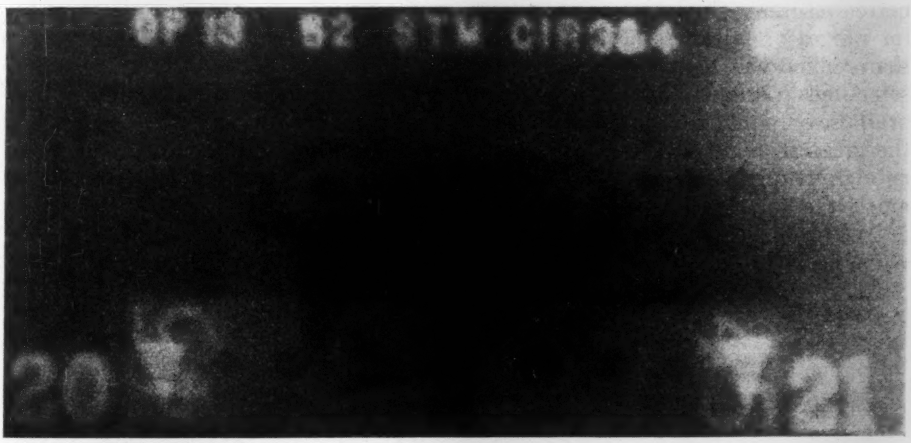

FIG. 7.—Fine-Grained Film Exposed by 1000 kv.p. X-rays.

This film development has, in the author's opinion, been as significant an advancement in the radiographic examination of heavy welded boiler drums and pressure vessels as has been the million-volt machine itself. Such a film, because of its slowness, is relatively insensitive to the longer wave length secondary radiation and, therefore, we might consider it a type of the ideal grid described above. Besides the use of fine grain film for million-volt X-rays, the best radiographs (of any plate thickness) are those obtained with a combination of the right type film and the right X-ray wave length or voltage, no grid or other such device being necessary. There are now on the market several grades of X-ray no-screen film each differing from the other as to graininess and speed. At plate thicknesses where secondary radiation and reasonable exposure times offer no problem the fastest of these films may be used with shorter wave lengths. As the thickness increases and secondary radiation becomes a problem the better technique is found in using a slower film at a voltage range high enough to give a reasonable exposure time. It has been found, however, that the best results are obtained by not extending the voltage range higher than necessary. For example, 400-kv.p. X-rays will give better results with the proper film and for ranges of thickness of 1¾ to 2¼ in. than million-volt X-rays will give. It is sometimes possible to filter X-ray beams or to use only that part of the beam from the greatest angle off the target of low or high-voltage equipment and achieve much the same results as though one had available the various ranges of X-ray equipment.

We are of the opinion that these new developments warrant the following changes in X-ray technique in the boiler shop:

1. That the Bucky grid need no longer be considered an essential part of X-ray technique.
2. That calcium tungstate screen exposure of X-ray film may be discarded.
3. That proper voltage and film combinations are essential for good radiographs of fixed ranges of plate thicknesses.
4. That finer grain film makes possible the successful use of shorter wave lengths, even shorter than those of the million-volt apparatus. Gamma rays give excellent results.
5. That X-ray techniques may now be standardized, and, if followed, satisfactory results are assured.

DISCUSSION

Mr. C. A. Adams.[1]—Is there any quantitative connection, which could be stated roughly, between the optimum voltage and the thickness? I understand that under ideal circumstances, using modern films, neither intensifying screens nor Bucky grids are desirable.

Mr. O. R. Carpenter.[2]—I have done some work along that line, but I would hesitate to put it into any concrete form. We do know that from our former experience we were more or less restricted because the type of film we used was a fast film primarily for use with calcium tungstate screens. Now as we have developed our techniques and as the no-screen film has come into general use, we have found that at particular ranges of voltage and for specified plate thicknesses, a particular no-screen film will give the best results in detail and contrast. This consequently led us to accept a number of types of films for standardized technique. To make a list offhand of that work would be impossible at this time. However, there is a very definite relationship, and I think it should be further worked out.

Mr. Adams.—On the assumption that you have an appropriate voltage for a thin plate, how does the time of exposure compare for, let us say, a $\frac{1}{2}$-in. plate at 200 kv., and a 4-in. plate at 1,000,000 v.?

Mr. Carpenter.—The time at 1,000,000 v. for a 4-in. thick plate could be very easily cut down to 3 to 4 min. That would be a very long exposure for a $\frac{1}{2}$-in. plate X-rayed at 200 kv. However, we used to take 30 sec. for a $\frac{1}{2}$ in. thick plate and 5 hr. for a $4\frac{1}{2}$ in. thick plate, which would give an exposure chart curve with a very steep slope. Nowadays the slope would change and we would have a very easy slope coming down from, say, 5 min. to a matter of one-half to one minute.

Mr. Adams.—At what thickness should you ideally increase the voltage above 1,000,000 v.?

Mr. Carpenter.—At the present time we have done work up to 6 in. in thickness. These exposures with the million-volt machine are running about 20 min., and that is not with the fine-grained film. Around 6 in. thick, we have to use a coarse-grained speedy film for that exposure time. That work happens to be with castings, and we were not interested in as full detail as we would have been if it had been a weld. If it had been a weld, I venture to say that the exposure time would have gone to twice or perhaps three times this because of the desire to use more sensitive film, which would have been beyond the practical limits of the machine. Therefore, if we were to make welds 6 in. or more thick, in all probability we would want to go to a higher voltage machine. At the present time, where we are working with welds up to 6 in. thick, I see no need for it.

Mr. Otto Zmeskal.[3]—Mr. Lorentz told us of using the $\frac{1}{4}$-in. lead sheet between the plate and the film. Is that necessary even when you use the $\frac{1}{4}$-in. filter right at the target?

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[1] Professor of Engineering, Emeritus, Harvard University, Cambridge, Mass.; and Consulting Engineer, Edward G. Budd Manufacturing Co., Philadelphia, Pa.

[2] Engineer, The Babcock & Wilcox Co., Barberton, Ohio.

[3] Assistant Professor of Metallurgy, Illinois Institute of Technology, Chicago, Ill.

MR. CARPENTER.—When we first started with 1000-kv. X-rays we used a $\frac{1}{4}$-in. filter at the target itself and also a filter at the film. As we went along we decided that the filter at the target was unnecessary, and we have gone to a 0.090-in. filter at the film only. The filter at the tube seemed to do very little good.

MR. W. J. SHANEMAN.[4]—Is the fine-grained film that you mentioned of the no-screen type?

MR. CARPENTER.—Yes, it was entirely no-screen type film. I think perhaps Mr. Seemann can explain more fully than I can what the nature of the film is.

THE CHAIRMAN (*Mr. Herman E. Seemann*[5]).—I think it has been said adequately that in studying the nature of radiographic film, attention must be paid to the assumed requirements of that film. If you are expecting to use a film with calcium tungstate intensifying screens or similar screens, you want a film which has a good sensitivity to blue light, and, relatively speaking, you do not care about its direct sensitivity to the X-rays, because you get a factor of reduction of 10 to 100 or even more in the exposure time with fluorescent screens.

If you consider the film for use with direct X-rays or with lead foil in contact with the film, you then pay attention to the response to the direct X-rays only. Generally speaking, the screen film is not very good for direct X-ray work, and *vice versa*.

MR. SHANEMAN.—How does that fine grain you mention compare in speed with the no-screen type?

MR. CARPENTER.—The easiest way I have to tell you that would be by means of reference to the Radium Chemical Co. exposure meter, which gives you a factor of 0.7 for standard no-screen film. If you use the fine-grained film, which we use altogether for gamma-ray work and higher voltage X-rays, the factor goes up, I believe, to 2.

MR. SEEMANN.—Perhaps I should make one more remark in connection with films. You will notice that in using radiographic film over a wide range of kilovoltages, the greater the thickness of material and the higher the voltage, the more grainy does the image appear. That, of course, is disturbing to the diagnostic use of the film, and therefore the use of a finer-grained material has definite advantages. There are so many applications that one cannot make a recommendation of film type without considering the individual problem.

MR. ADAMS.—How high can you go with the present film you have? Do you think that would be good for 2,000,000 v.?

MR. SEEMANN.—I see no reason why it should not be, but we have not tried it at that voltage.

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[4] Westinghouse Electric and Manufacturing Co., South Philadelphia Plant, Lester, Pa.

[5] Physicist, Eastman Kodak Research Laboratories, Eastman Kodak Co., Rochester, N. Y.

THE GAMMA-RAY RADIOGRAPHY OF WELDED HIGH PRESSURE POWER PLANT PIPING

By R. W. Emerson[1]

Synopsis

This paper discusses the apparatus necessary, and the procedure used in the gamma-ray radiography of both circumferential and longitudinal seams in welded piping. Also discussed is the effect of size of radiant source, distance from source to film, metal thickness, and type of film on the resultant radiographic sensitivity.

An accurately drilled test block, having three series of holes of $\frac{1}{16}$, $\frac{1}{8}$, and $\frac{3}{16}$-in. diameters, and depths of 0.010 to 0.100 in. in steps of 0.010 in., was radiographed using many combinations of source to film distance and metal thickness, using both a 25- and 100-mg. radium source. The smaller source definitely proved superior to the larger for short source-to-film distances but offered little if any advantage as the source-to-film distance increased beyond 6 in.

Two circumferential test pipe welds were made in which many defects, common to welded piping, were purposely made. These welds were first radiographed and then sectioned in many places, polished and etched and used to check the actual defect with the radiograph.

Two types of penetrameters were placed on both the source side and film side of the pipe welds. It is the author's opinion that there is little if any practical difference in the results obtained with the penetrameters in the two positions (at least for wall thickness up to 1.5 in.). It is agreed, however, that penetrameter detail is slightly better when the pentrameters are placed on the film side, which of course does not give an absolutely true picture of the detail of a defect comparable to the size of the penetrameter holes which is close to the root of the weld.

Also discussed and illustrated are a few of the various types of piping radiography encountered in the piping industry.

In view of continual rise of boiler pressures and temperatures, due to the development of new alloys, together with the development of the shielded-arc welding electrode and advancement in the art of welding, rigid specifications and inspection must be met on power-plant materials and their subsequent fabrication. In this the continual development and application of both X-ray and Gamma-ray radiography as non-destructive methods of inspection of metallurgical materials has played an important part.

The commercial importance of the use of radium emanation as a means of determining the soundness of metallurgical materials was first recognized in 1930 by Mehl, Doan, and Barrett (**1**).[2] Today, radiographic inspection of welded joints in certain boiler and main steam piping is mandatory under the rules of the A.S.M.E. Power Boiler Code.

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[1] Metallurgist, Pittsburgh Piping and Equipment Co., Pittsburgh, Pa.

The gamma-ray method is believed preferable to X-ray when radiographing welded piping, due to the relative simplicity and greater adaptability of this method; particularly is this true when field radiography is required.

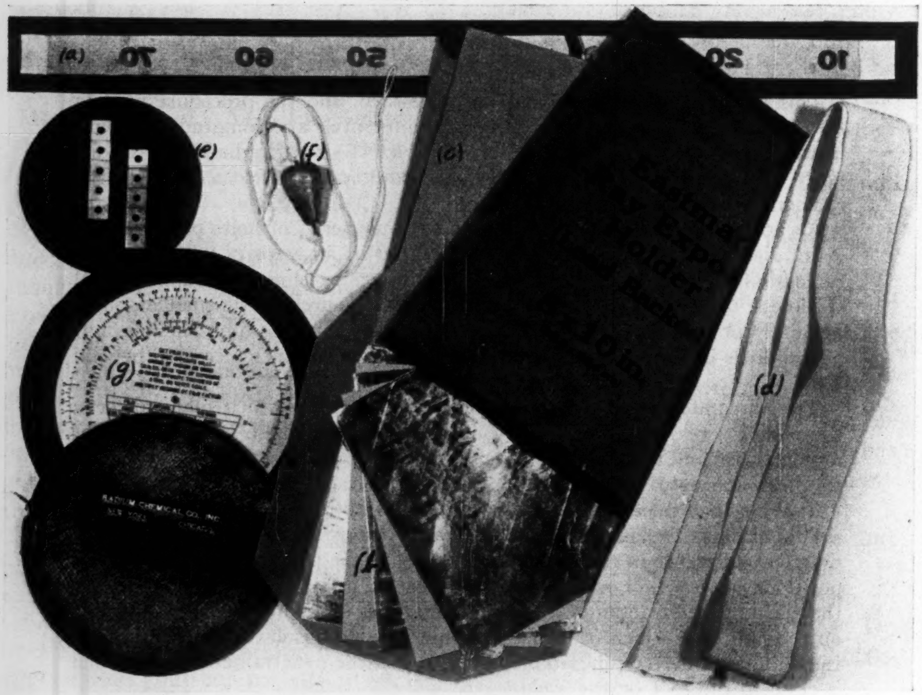

FIG. 1.—Apparatus Required for Gamma-Ray Radiography of Welded Pipe.

The A.S.M.E. Power Boiler Code requires radiography of fusion welded joints in (1) pipes or tubes and superheater headers exceeding 16 in. nominal pipe size or 1$\frac{5}{8}$ in. in wall thickness that are to contain steam, and pipes or tubes exceeding 10 in. nominal pipe size or 1$\frac{1}{8}$ in. in wall thickness that are to contain water when in each case the welds are not to be in contact with furnace gases; (2) in pipes or tubes exceeding 6 in. nominal pipe size or $\frac{3}{4}$ in. wall thickness when the welds are to be in contact with furnace gases but not subject to radiation from the furnace; and (3) in pipes or tubes exceeding 4 in. outside diameter or $\frac{1}{2}$ in. wall thickness when the welds are to be in contact with furnace gases and subject to radiation from the furnace.

In some cases, individuals have requested radiographic inspection of welded high-pressure piping which has not been within the scope of the Power Boiler Code. This is believed primarily to be due to the increased severity of service conditions placed upon power-plant piping.

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[2] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 1129

APPARATUS AND PROCEDURE

With the possible exception of a special jig or fixture for holding the radium capsule (gamma-ray source), the apparatus required is essentially as shown in Fig. 1.

Lead numbers are placed on adhesive tape (*a*) and in turn placed around the circumference or along the seam of the welded joint to be radiographed for the purpose of identifying the location of any possible defect registered on the film. Three sheets of 0.005 in. lead foil are alternated with two films (*b*) and placed in a light-tight film holder (*c*). The film holder or holders (cassettes) are then strapped around the circumference or along the seam to be radiographed by means of 2-in. wide elastic banding (*d*). Penetrameters or sensitivity gages (*e*) are placed adjacent to, but at the extremities of the portion of the joint undergoing exposure. These gages whenever practicable, must be placed on the opposite side of the joint from the film.[3] With the film in position, the radium capsule (*f*) is placed in position at a suitable distance on the side of the joint opposite to that from the film.

Since radium continuously emanates gamma rays, exposure begins immediately upon placement of the radium into exposure position. The time of exposure is dependent upon the quantity of radium being used, the distance from radium to film, the thickness of the steel being radiographed, and the film speed. These factors being known, the time of exposure is calculated by use of the exposure meter (*g*).

There are at least four reasons for the use of the double-film technique as above described.

1. By slightly underexposing the film for single viewing and then viewing the two films superimposed, an increase in contrast is believed to be obtained over that by a longer exposure using single film.

2. Simultaneously with increased contrast, which is highly desirable in gamma-ray radiography, a reduction in exposure time using double film is accomplished.

3. Any variation in film density resulting from defective film or improper developing can be readily checked, by checking one film against the other, it being relatively certain that unless variation in film density is coincident when the two films are viewed superimposed, such variations would not be the result of defective material undergoing inspection.

4. The use of double film provides, if necessary, a record for both the fabricator and the customer, though generally

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[3] *Code Interpretation Case No. 953:*

Inquiry.—The Code states that when radiographing welded joints the penetrameters must be placed on the radiation side of the work. When radiographing a circumferential pipe joint by placing a radium capsule inside of the pipe, it is often very difficult and expensive to place the penetrameters inside of the pipe. Is it permissible in such cases to place the penetrameters on the outside or film side of the joint under examination?

Reply.—In the situations cited in the inquiry it is the opinion of the committee that the penetrameters may be placed on the film side of circumferential pipe joints, provided the following additional requirements are satisfied:

1. A preliminary radiograph shall be made with a short piece of pipe with penetrameters on both the inside and outside. The diameter of the pipe employed in making this proof radiograph shall be the same as that of the job in hand and its wall thickness shall be equal to that of the over-all thickness of the joint to be radiographed, including both backing ring and reinforcement if these are present in the joint to be examined. The radium capsule employed in making this proof radiograph, together with all other items of technique such as the location of the capsule and the time of exposure, shall be the same as employed on the actual job. Each penetrameter shall be provided with a marker which will show up clearly on the film and which will indicate the side of the joint on which it is located: "F" for the film side and "R" for the radiation side.

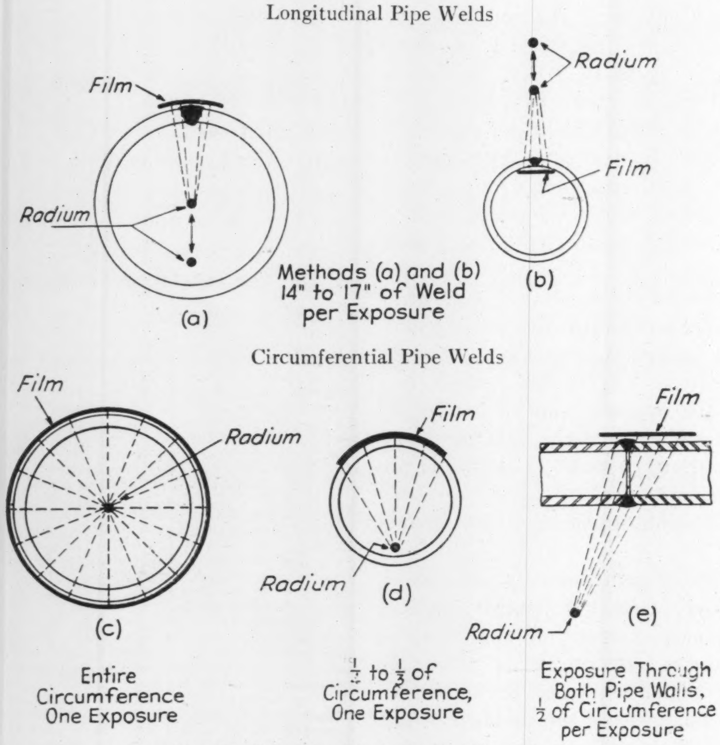
2. This proof radiograph together with the radiograph of the joint in question shall be submitted to the inspector who will then decide as to the efficiency of the radiograph of the actual joint with the penetrameter on the film side. Each such production penetrameter shall be provided with the "F" marker referred to in 1.

3. If both of these radiographs are satisfactory from the standpoint of penetrameter definition and contrast, the Code requirements for the minimum source distance, and for the minimum ratio of source distance to film distance, may be waived but the ratio of source distance to film distance shall be clearly indicated on each production film.

4. When the radium capsule is placed on the axis of the joint and the complete circumference radiographed with a single exposure, four penetrameters uniformly spaced shall be employed.

both sets of films are filed by the fabricator.

The procedure followed in placing the radium and film will depend primarily on whether the pipe weld undergoing inspection is longitudinal or circumferential; in the case of either the placement will further depend upon both the pipe diameter and wall thickness. When making a radiographic setup, the quality of the resulting radiograph must first be considered, and second, the time involved to obtain the desired results.


Longitudinal Pipe Welds

Film
Radium
(a)
Radium
Film
(b)
Methods (a) and (b)
14" to 17" of Weld
per Exposure

Circumferential Pipe Welds

Film
Radium
(c)
Entire
Circumference
One Exposure
Film
Radium
(d)
1/4 to 1/3 of
Circumference,
One Exposure
Film
Radium
(e)
Exposure Through
Both Pipe Walls,
1/2 of Circumference
per Exposure

FIG. 2.—Exposure Methods for Pipe Welds.

Several procedures used in setting up for radiography of both longitudinal and circumferential seams are shown in Fig. 2. Figure 2 (*a*) and (*b*) illustrate the methods used in radiographing longitudinal pipe seams. Method (*a*) is usually used when the nominal pipe diameter exceeds 10 in. and method (*b*) used when the diameter is less than 10 in. The length of seam normally radiographed at one exposure is 1 to 1½ times the perpendicular distance from the radium to film, the factor of 1 being used when the pipe wall thickness is 2 in. or greater, and this factor increasing to 1½ for a wall thickness of 1 in. or less (2). For longitudinal welded seams in which the wall thickness does not exceed 1 in. and the radium capsule does not exceed 100 mg. an optimum length of seam to radiograph at one exposure would be 14 to 17 in., using a radium to film distance of 9 to 12 in, respectively. By doubling the radium-to-film distance, the length of seam radiographed at one exposure could likewise be

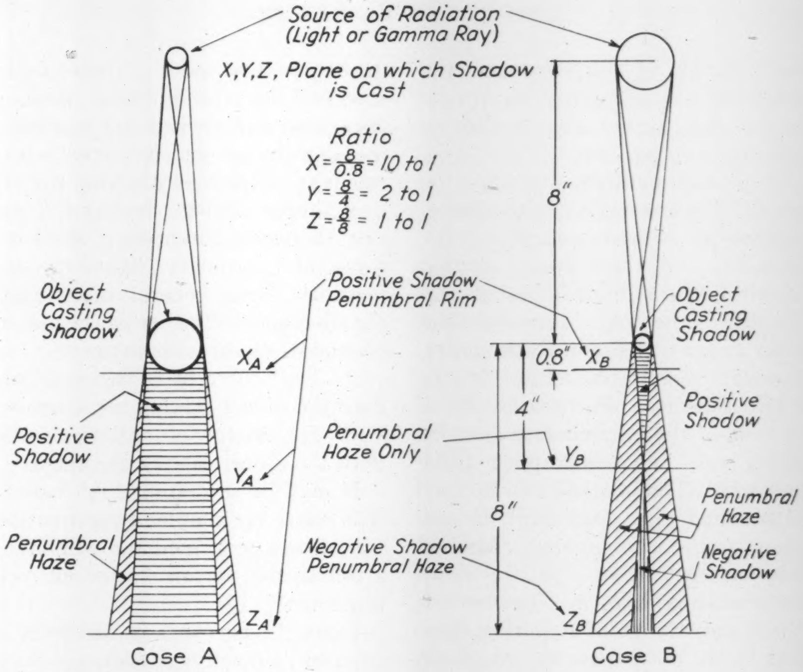
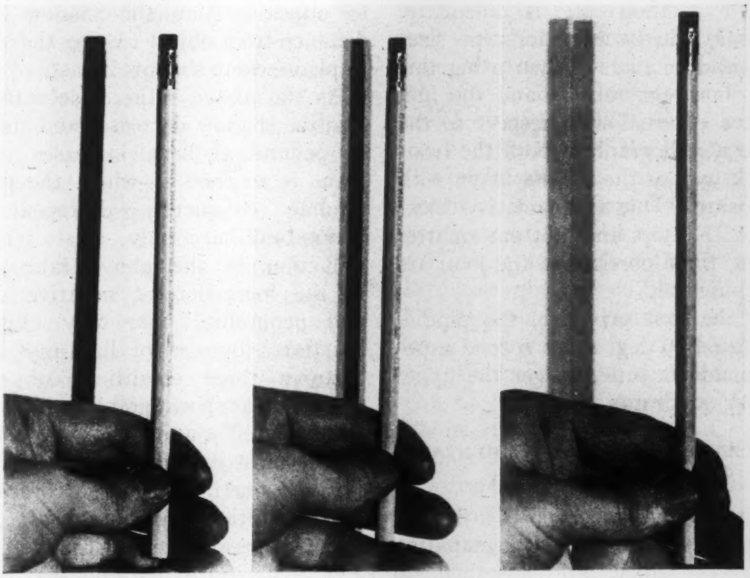

Source of Radiation
(Light or Gamma Ray)
X,Y,Z, Plane on which Shadow is Cast
Ratio
$X=\frac{8}{0.8}=10$ to 1
$Y=\frac{8}{4}=2$ to 1
$Z=\frac{8}{8}=1$ to 1
Object Casting Shadow
Positive Shadow Penumbral Rim
X_A
Positive Shadow
Y_A
Penumbral Haze
Z_A
Case A
8"
Object Casting Shadow
0.8"
X_B
4"
Positive Shadow
Penumbral Haze Only
Y_B
8"
Penumbral Haze
Negative Shadow Penumbral Haze
Negative Shadow
Z_B
Case B

FIG. 3.—Schematic Diagram of Shadow Formation (After Doan (3)).

Case A—Source of radiation smaller than the object casting the shadow.
Case B—Source of radiation larger than the object casting the shadow.



(*a*) Positive shadow only, see plane X_A, Fig. 3.

(*b*) Positive and penumbral shadow, see plane X_B, Fig. 3.

(*c*) Penumbral shadow only, see plane Y_B, Fig. 3.

FIG. 4.—Light-Ray Shadow Formations Corresponding to Diagrammatic Conditions of Fig. 3.

doubled. By doing this, however, the exposure time is quadrupled, thus resulting in an ultimate net loss in time of approximately 50 per cent.

Three general methods of placing the radium and film are applicable to circumferential welds as shown in Fig. 2 (*c*), (*d*), and (*e*). Of these three, method (*c*), in which the radium is placed centrally within the pipe and the film wrapped around the entire circumference, is far superior to either of the other two schemes. Fortunately 90 to 95 per cent of the circumferential welds in piping can be radiographed using this method. This method can be used on all nominal pipe sizes down to and including 6 in. in diameter. Method (*d*) may be used on all pipe sizes down to and including 4 in. in diameter but has the disadvantage of requiring three or four separate exposures which in addition to the added exposure time require additional setup time and therefore would not be used on any pipe size in which method (*c*) is adequate. Method (*e*) can be used for pipe sizes of 4 in. and smaller. When using this method, both the radium and the film are placed external with respect to the pipe and a radiograph of both the front and back wall of the pipe is taken with one exposure. Due to the relative thickening of the top and bottom quarter segments, these portions of the joint are underexposed.

After the first exposure, the pipe is then rotated 90 deg. and a second exposure is made in order to get the upper and lower quadrants.

A DISCUSSION AND EXPERIMENTAL STUDY OF SENSITIVITY

A gamma-ray radiograph is in reality a shadow picture produced by emanating gamma-rays which are absorbed to various degrees depending upon the thickness of the metal through which the rays must pass. Those rays not absorbed by the material undergoing inspection are picked up by the film, the density of which varies with the intensity of the rays reaching the film.

A simple analogy may be shown to exist between gamma-ray shadow pictures and ordinary light-ray shadow pictures. Two general cases exist, in the discussion of either of the two types of shadow pictures, as follows:

A. The source of radiation is smaller than the object casting the shadow.

B. The source of radiation is larger than the object casting the shadow.

It may be seen from Fig. 3 that case *A* is relatively simple; a positive shadow will always be formed and will have only a relatively narrow fringe of penumbral haze.

Case *B*, however, is decidedly more complex; a positive shadow accompanied by a narrow band of haze can only be obtained by having a relatively large ratio of the distance from radiant source to object casting the shadow to the distance from object casting the shadow to plane where shadow is cast.

As the above ratio is decreased the positive shadow decreases and the band of penumbral haze increases until a plane is reached at which the positive shadow completely disappears and penumbral haze only, exists. Further reduction in the above ratio results in the formation of negative shadow and penumbral haze only. Figure 4 illustrates by use of light-ray shadow pictures three conditions of shadow formation as shown graphically in Fig. 3, planes X_A, X_B, and Y_B.

The most desirable shadow picture is logically one in which high contrast and good definition can be obtained. Figure 4 (*b*), corresponding to plane X_B of Fig. 3, illustrates the fact that in order to obtain the above qualities, when the source of radiation is larger than the object casting the shadow (case *B*), a relatively large source-to-film ratio must be maintained,[4] since these qualities are dependent upon a positive shadow with little or no penumbra. On the contrary, however, if the source of radiation is smaller than the object casting the shadow (case *A*), the need for maintaining a large source-to-film ratio is much less critical since a positive shadow with a relatively small penumbra will always be obtained.

The simplest and most logical method of radiographing circumferential pipe welds, as previously mentioned, is that of placing the radium on the circumferential center line as shown in Fig. 2 (*c*). It is to be noted that the source-to-film ratio when using this method, becomes automatically fixed. Since it is desirable to locate the smallest possible defects and since a positive shadow accompanied by a minimum of penumbra is further desirable, it becomes clear that the size of the source of radiation must be reasonably near if not equal in size to the smallest defect which is expected to be found, unless of course a reasonably large source-to-film ratio can be maintained. If this ratio is small, however, and the flaw diameter (taken perpendicular to the path of the rays) is small with respect to the radiant source; the true shadow will then be convergent and may be entirely lost to the film. The negative and penumbral shadows are, however, divergent and while Doan has shown penumbral shadow registry of flaws, this is not believed to be too reliable for flaw depths as small as $\frac{1}{32}$ in. with flaw diameters of approximately the same magnitude since the variation in density between the penumbral shadow of such a small defect and the adjacent film would be very small (3). Furthermore, defects producing a given low contrast become increasingly hard to locate as the size of the defects decrease.

In discussing the size of defects observable (sensitivity), the question might be asked, "What is sensitivity and how is it defined?" In the opinion of the author, sensitivity is the degree to which the process is capable of detecting flaws. It is further the author's opinion that 2 per cent sensitivity which is the established requirement of the A.S.M.E. Power Boiler Code, does or should mean that the process must be capable of detecting a defect which has a depth, parallel to the path of the rays, of 2 per cent of the plate thickness and a diameter of equal size measured perpendicular to the path of the rays. Whether such defects have been picked up, if present, is determined by the observation of a sensitivity gage or penetrameter which is placed on the steel being radiographed. The latest type of penetrameter (not shown in Fig. 1) is a single thickness strip of mild steel $\frac{1}{2}$ by $1\frac{1}{2}$ in. and must have a thickness of 2 per cent of the plate thickness. This strip contains three holes having diameters of 2, 3, and 4 times the strip (penetrameter) thickness. The A.S.M.E. Code states that the image of all three holes shall be visible. This means that it should be possible to detect a defect having a depth of 2 per cent of the plate thickness and a diameter of 4 per cent of the plate thickness (smallest hole in penetrameter).

Since the penetrameter itself is large with respect to the gamma-ray source, a positive shadow is cast on the film and, according to Fig. 3, case *A*, the penetrameter image will be seen. The holes in the penetrameter are sufficiently small with respect to the source in some cases so

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[4] Source-to-film ratio when mentioned hereafter, refers to the ratio

$$\frac{\text{Distance from source of radiation to object casting the shadow}}{\text{Distance from object casting shadow to plane on which shadow is cast}}.$$

that the positive shadow cast by these holes converge to a point and therefore cast only a hazy penumbral shadow, if a shadow is cast at all.

It is for this reason that the outline of the penetrameter can be readily detected when at times the small penetrameter holes are difficult to pick up. If the outline of the penetrameter can be picked up on the film but the penetrameter holes are not visible, this indicates that the source-to-film ratio is too small or the size of the source is too large or perhaps both.

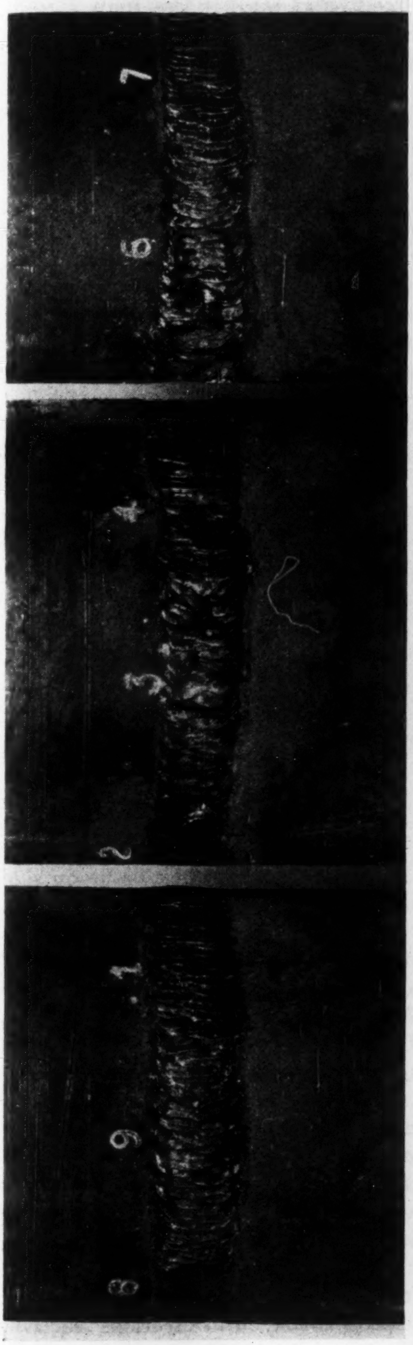
TABLE I.—ABSOLUTE RADIOGRAPHIC SENSITIVITY AS EFFECTED BY SOURCE-TO-FILM DISTANCE, METAL THICKNESS, AND SOURCE INTENSITY.

Exposure	Radium Capsule, mg.	Distance, in.		Ratio $\frac{A}{B}$	Depth of Last Hole Visible, in.			Comments
		(A) Source to Plane of Holes	(B) Plane of Holes to Film		$\frac{1}{16}$-in. Hole	$\frac{1}{8}$-in. Hole	$\frac{3}{16}$-in. Hole	
No. 1	25	1.75	0.50	3.50	0.01	0.01	0.01?	Hazy—0.01 very faint
No. 2		3.50	0,50	7.00	0.01	0.01	0.01	Better detail than No. 1—0.01 very faint
No. 3		3.50	1.00	3.50	0.02?	0.02?	0.02?	Hazy—enlargement of image of holes
No. 4		5.25	0.50	10.50	0.01?	0.01	0.01	Sharp image—0.01 very faint
No. 5		5.25	1.00	5.25	0.02	0.02	0.02	Sharp image—slight enlargement
No. 6		5.25	1.50	3.50	0.03	0.03	0.03	Very hazy—enlargement of image
No. 7		7.00	0.50	14.00	0.01?	0.01	0.01	Extremely sharp image—0.01 faint
No. 8		7.00	1.00	7.00	0.03	0.02	0.02	Sharp image
No. 9		7.00	1.50	4.67	....	....	....	Underexposed
No. 11	100	1.75	0.50	3.50	0.02	0.02	0.02	Much more hazy than No. 1
No. 21		3.50	0.50	7.00	0.02	0.02	0.02	Hazy but improved over No. 11
No. 31		3.50	1.00	3.50	0.03	0.03	0.03	Very indistinct and hazy
No. 41		5.25	0.50	10.50	0.02	0.01	0.01	Sharp image—good detail
No. 51		5.25	1.00	5.25	0.03	0.02	0.02	Hazy image—0.02 faint
No. 61		5.25	1.50	3.50	0.03?	0.03	0.03	Very hazy and enlarged image poor—0.03 faint.
No. 71		7.00	0.50	14.00	0.01?	0.01	0.01	Sharp image—good—0.01 faint
No. 81		7.00	1.00	7.00	0.03	0.02	0.02	Hazy but fair detail—0.02 faint
No. 91		7.00	1.50	4.67	0.03	0.03	0.03	Fair detail—0.03 faint

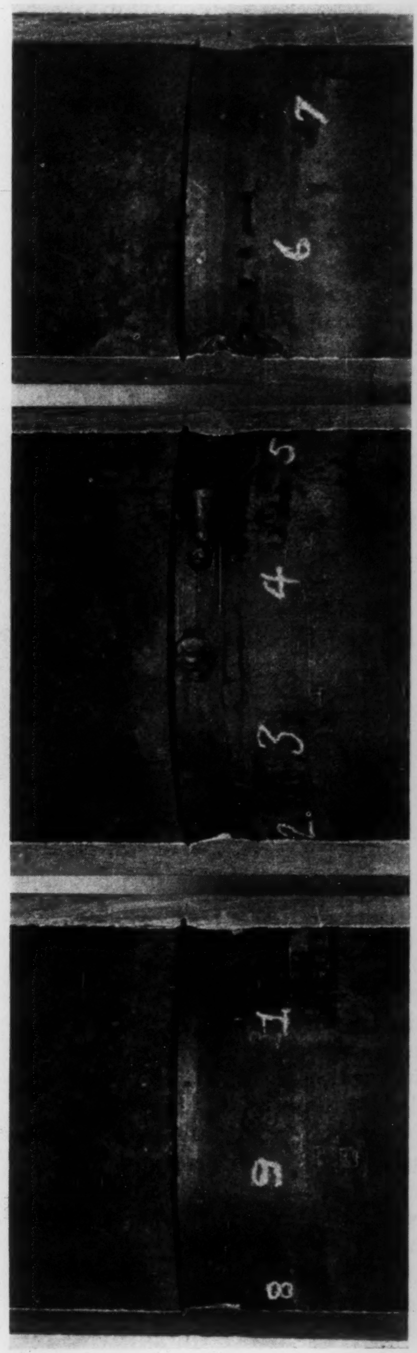
Radiographic sensitivity as indicated by the penetrameter image is to a very large extent dependent upon the type of film used. When using a fast coarse-grain film having a 20 per cent slide rule factor the limiting absolute sensitivity has been found to be about 0.02 in. This limiting sensitivity was found to hold true for source-to-film ratios as high as 25 to 1 with wall thickness as low as $\frac{3}{8}$ in. This results in a relative sensitivity of 2 per cent or better for respective wall thickness of 1 in. or slightly greater, with a rapidly diminishing sensitivity of as low as 5.3 per cent for $\frac{3}{8}$-in. thick material. This is in line with the results of Norton and Ziegler (4) and later referred to by Mochel (5).

The use of a more recently developed fine-grain film having a 150 per cent slide rule factor has been found to pick up readily four of the five steps of the type of penetrameter shown in Fig. 1. With this film, the limiting absolute sensitivity has been found to be about 0.01 in. This has been found to hold true, as in the above case, for ratios as high as 25 to 1 and wall thickness as low as $\frac{5}{16}$ in. With a limiting absolute sensitivity of 0.01 in. it is therefore possible with this type of film to obtain a relative sensitivity of 2 per cent on material thickness as low as $\frac{1}{2}$ in. as contrasted with 1-in. thickness using the higher speed film.

The source intensity is related to the size of the source; the greater the intensity, the larger the source. It there-



(*a*) Outside of pipe weld.



(*b*) Inside of pipe weld.

FIG. 5.—Experimental Pipe Weld, $6\frac{5}{8}$ in. in outside diameter by 0.432 in. in wall thickness.

fore becomes obvious in view of previous discussion that in order to obtain equal results, it is necessary to use increasingly larger source-to-film ratios with increasingly larger source intensities. The results shown in Table I are believed to illustrate this fact.

Table I gives the results of a study in which a $\frac{1}{2}$ in. thick machined steel block was drilled with three series of holes having diameters of $\frac{1}{16}$, $\frac{1}{8}$ and $\frac{3}{16}$ in. and depths ranging from 0.010 to 0.100 in. in steps of 0.010 in. Two additional $\frac{1}{2}$-in. blocks were machined and used to build up the steel thickness to $1\frac{1}{2}$ in. Various source-to-film ratios were used as indicated in Table I and the depth of the shallowest hole visible is listed. A 25-mg. radium source was used in exposures Nos. 1 to 9 and a 100-mg. source was used in exposures Nos. 11 to 19. A high-contrast - fine-grain film having a 150 per cent slide rule factor was used in all exposures.

In contrasting exposure Nos. 1, 2, and 3 with respective exposures Nos. 11, 12, and 13, it can be seen that the results of the former three, in which the 25-mg. source was used, are definitely superior to the latter. As the source-to-film ratio is increased, however, it may be seen that superiority of the smaller capsule diminishes, though it may be said that for exposures in which the source-to-film ratio is less than 7 to 1, a 50-mg. source should not be exceeded, with a 25-mg. capsule preferable.

AN EXPERIMENTAL STUDY AND RADIOGRAPHIC INTERPRETATION OF DEFECTIVE PIPE WELDS

Because of insufficient knowledge on the part of the author to properly interpret gamma-ray radiographic films, two experimental pipe welds were made in which many common pipe weld defects were purposely made in order that the resulting radiographs could be accurately checked with the actual defect by polishing and etching many sections actually cut from the defective weld.

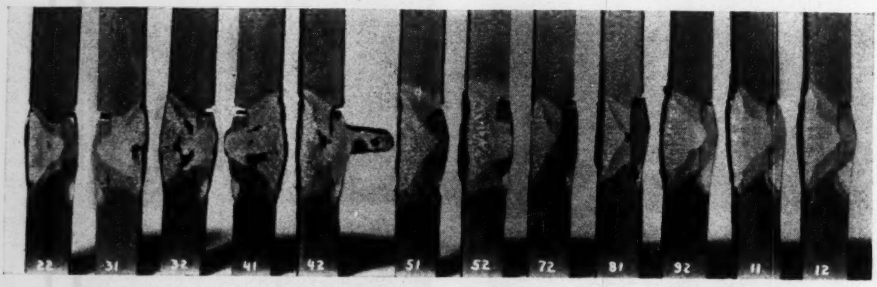


FIG. 6.—Cross-Sections of Weld Shown in Fig. 5. Etched in 15 per cent aqueous nitric acid (× 1). Reduced one half in reproduction.

In the first experiment a $6\frac{5}{8}$-in. outside diameter 0.432-in. wall pipe was used. A 100-mg. source was placed centrally within the pipe resulting in a fixed source-to-film ratio of approximately 5.7 to 1. Two Agfa Non-Screen films (20 per cent slide rule factor) were alternated between three sheets of 0.005-in. lead foil, placed in the cassette, and wrapped around the circumference of the weld.

After radiographing, the pipe was cut into three sections and both the outside and inside of the weld was photographed as shown in Fig. 5. Following this, the three sections were then cut into a total of twenty 1-in. sections each of which were polished and etched to show the various defects purposely produced. Twelve of these sections, shown in Fig. 6, are sufficient to illustrate a majority of difficulties which might be encountered. The radiograph of this weld is shown in Fig. 7. The exact location of each of the twelve sections of Fig. 6 with respect to the radiograph are so marked on Fig. 7.

Practically all pipe welds used in high-pressure power plant piping have a solid machined back-up ring so that good penetration can be secured without the possibility of having "icicles" protruding into the pipe. The pipe ends in practically all cases are so machined as to allow for a partial if not complete recess of such rings. Since there are many methods of inserting these rings with a still greater number of viewpoints as to width and thickness of rings, confusion in interpretation of radiographs are not unlikely. From Figs. 5 (*b*) and 6 it may be seen that the backing ring is recessed and seal welded to the pipe on one side.

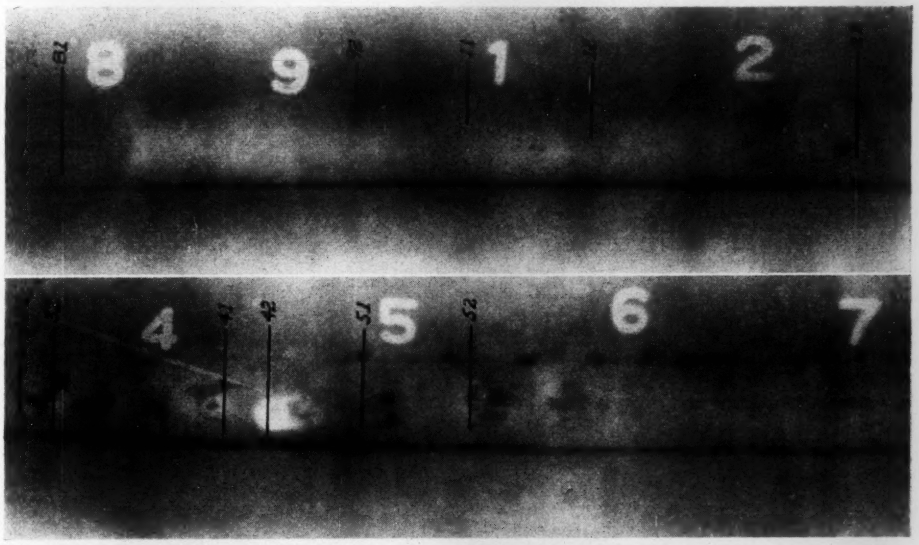
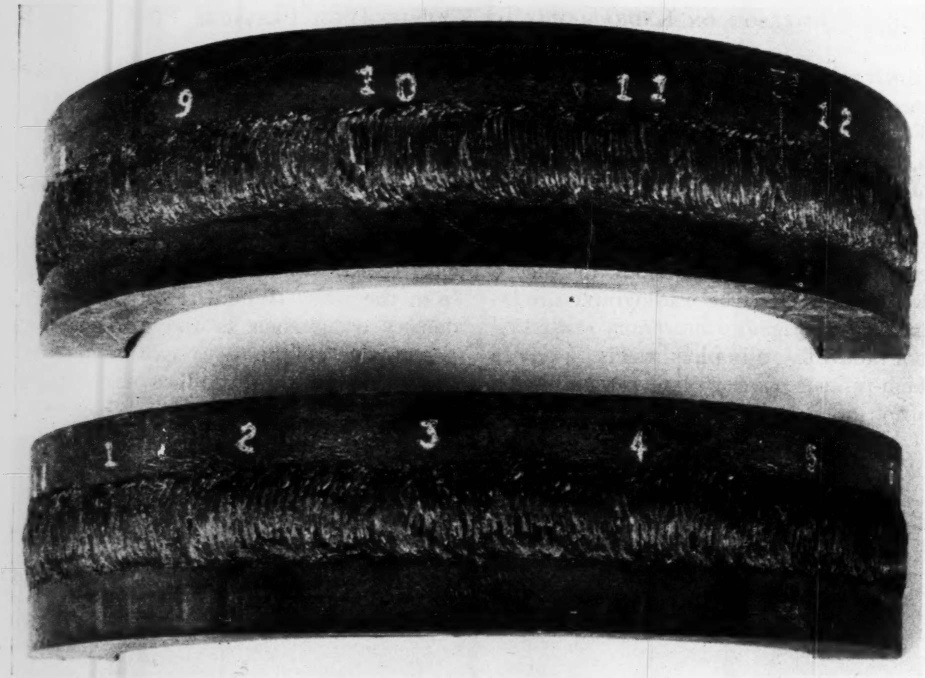


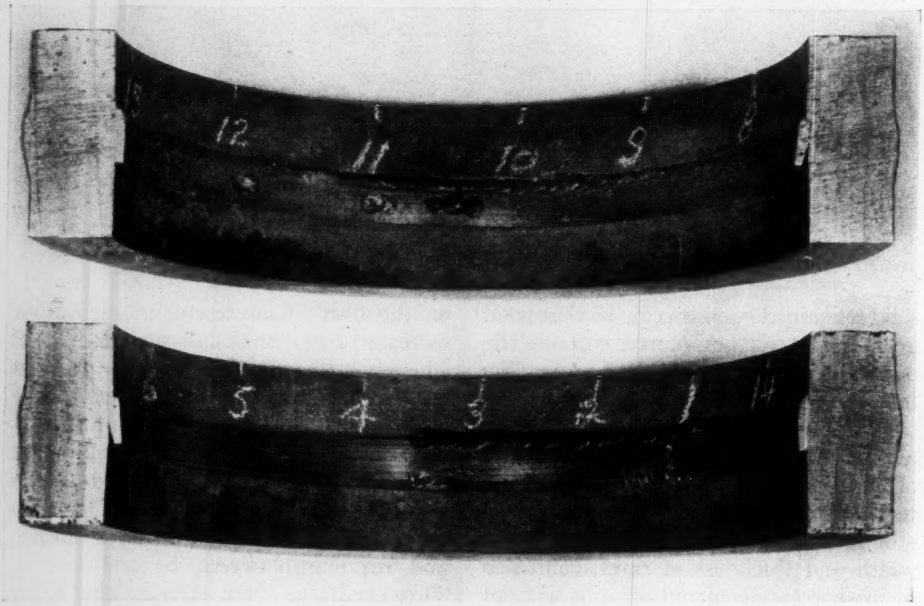
FIG. 7.—Gamma-Ray Radiograph of Experimental Pipe Weld (Fig. 5).

Sections Nos. 31, 32, and 41, Fig. 6, show large slag inclusions which resulted from welding over the top of and trapping slag. In addition, section No. 41 shows the backing ring to be melted with a small amount of metal protruding from the ring. Where regions of lesser density occur such as above, a greater percentage of the rays can penetrate the steel and thus produce dark spots on the film. Conversely, in regions of increased metal thickness such as No. 42 where an "icicle" has actually formed on the backing ring, light areas occur on the film.

Lack of penetration, or no welding at all, of the backing ring seal bead can be observed in Figs. 5 (*b*) and 6. This may be seen on the radiograph adjacent to, and running between, Nos. 2 and 8. This occurs as dark streaks and spots of varying density. Also observable on the radiograph is a dark streak running


9
10
11
12
1
2
3
4
5

(*a*) Outside surface.


15
12
11
10
9
8
6
5
4
3
2
1
14

(*b*) Inside surface.

FIG. 8.—Experimental Pipe Weld, 14 in. in outside diameter by 1.412 in. in wall thickness.

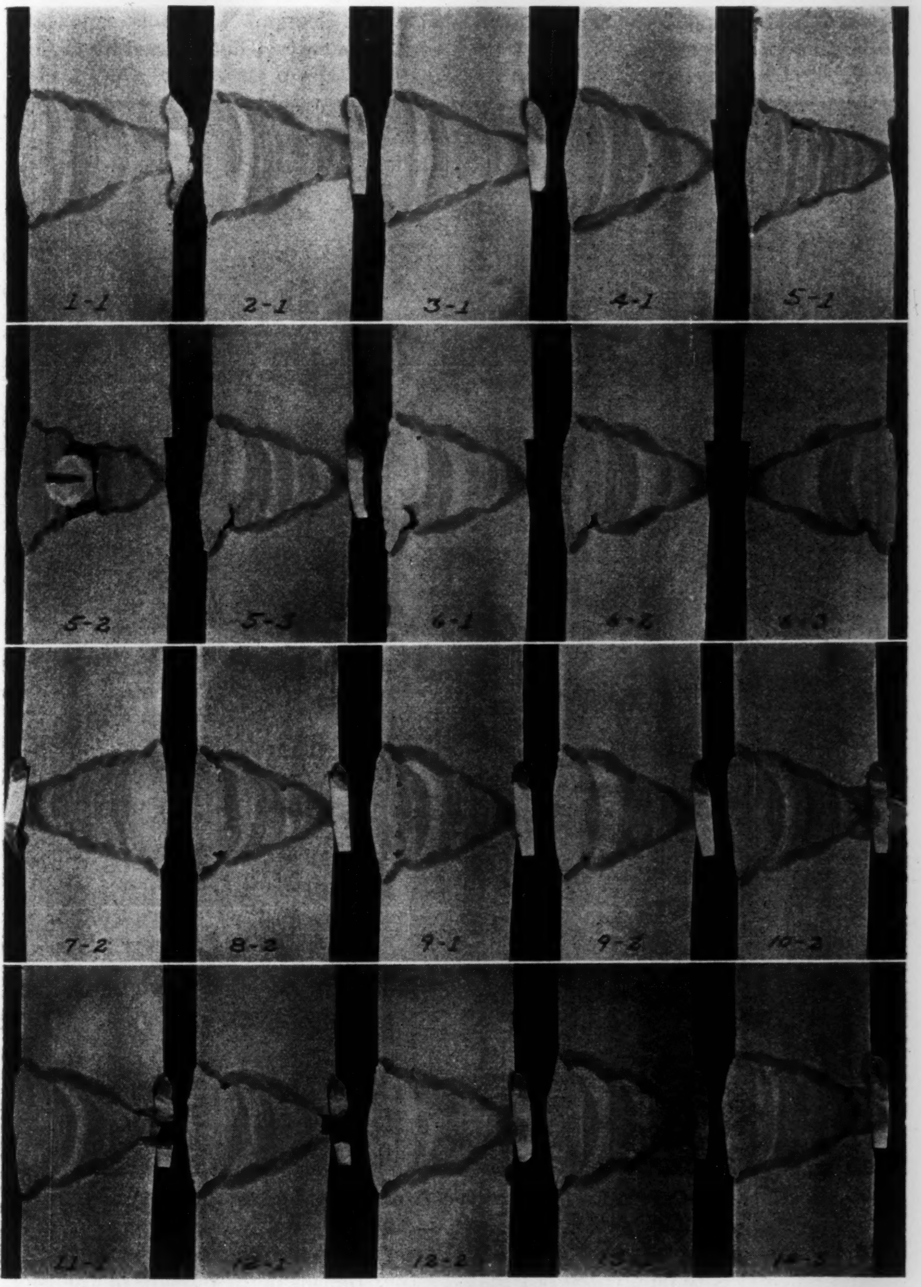

1-1
2-1
3-1
4-1
5-1
5-2
5-3
6-1
6-2
6-3
7-2
8-2
9-1
9-2
10-2
11-1
12-1
12-2
14-3

FIG. 9.—Cross-Sections of Weld Shown in Fig. 8. Etched in 15 per cent aqueous nitric acid (× 1). Reduced one half in reproduction.

directly through the center of the weld between Nos. 6 and 8. This is shown to be lack of penetration of the butt weld to the backing ring (Fig. 6, Nos. 72 and 81). It is to be pointed out that the condition of lack of penetration of both the seal bead and the butt weld as shown in section No. 81 is something which can only be determined by radiography and not by visual inspection of either the inside or outside of the pipe. One other condition which is to be ob-

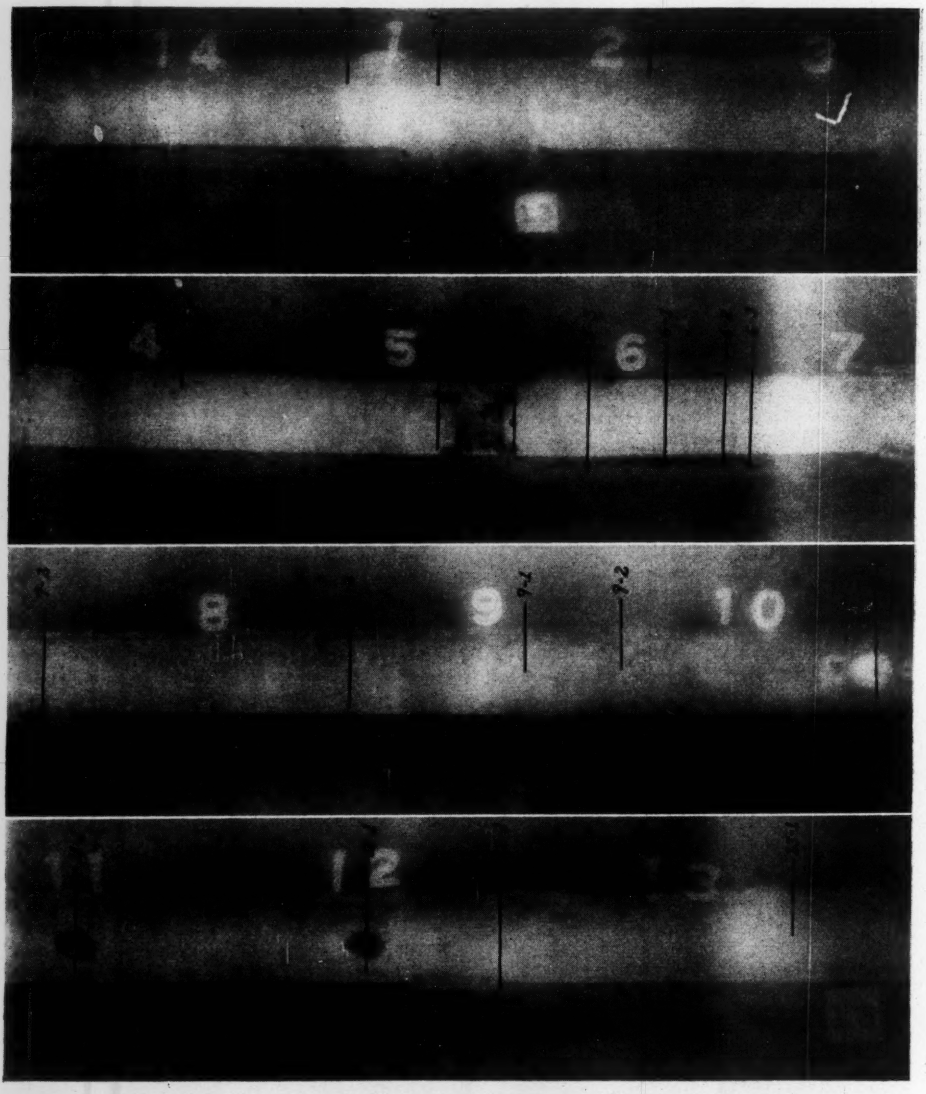


FIG. 10.—Gamma-Ray Radiograph of Experimental Pipe Weld (Fig. 8).

served on the radiograph is that of the change in density of the weld at No. 8. This is the result of a change in section from a flush weld bead to one of slight reinforcement, as noted from sections Nos. 81 and 92. Between Nos. 8 and 2 (on radiograph) an attempt was made to produce a weld which was satisfactory in all respects, sections Nos. 92, 11, and 12, of which are shown in Fig. 6. It may be seen from the radiograph, however, that a small gas pocket formed adjacent to No. 1.

In the second experiment, a 14-in. outside diameter 1.4-in. wall pipe was used. A 25-mg. radium source was placed centrally within the pipe resulting in a fixed source-to-film ratio in this case of approximately 3.7 to 1. For this exposure two Eastman Hi-Contrast Fine Grain Type A films (150 per cent slide rule factor) were alternated with three sheets of 0.005-in. lead foil and wrapped around the circumference of the pipe. The reason for using both the smaller radium capsule and the fine-grain film was due to the simultaneous decrease in source-to-film ratio and increase in wall thickness which is in line with the previous discussion on sensitivity. The experimental procedure of producing weld defects, followed by radiography and sectioning, was followed out as in the case of the first experiment.

Figure 8 shows the outside and inside of the weld. Figure 9 shows 20 out of a total of 42 sections which were polished and etched to reveal the actual defects. Figure 10 shows the defects as revealed by radiography prior to any sectioning.

It may first of all be recognized that the appearance of the entire cover pass shown in Fig. 8 (*a*), is misleading with respect to the many defects present in this weld, since the surface appearance of this weld would normally indicate one of good quality. Observation of the inside of the weld and backing ring, Fig. 8 (*b*), however, clearly indicates several unsatisfactory conditions. Melting of the backing ring and an "icicle" condition is seen between Nos. 10 and 11. Two large holes in the backing ring are present at Nos. 11 and 12. This resulted from excessive welding current when depositing the first bead. In the region of No. 7, the backing ring has pulled away from the pipe on one side. Also seen is scattered and inconsistent tacking of the backing ring to the pipe.

It must be kept in mind in many instances where radiography of field welds is required that the inside of the pipe is completely inaccessible in so far as visual observation is concerned, there being only a 1-in. hole drilled in the pipe adjacent to the weld for the insertion of the radium capsule. For this reason it is extremely important properly to distinguish minor defects, which may be in or adjacent to the backing ring, from major weld defects, which may be injurious to the piping system, without reference to the root side of the pipe weld.

Since the exact location of each of the 20 polished sections, Fig. 9, is marked on the radiographs, it is believed that the radiographic interpretation becomes essentially self explanatory.

With reference to the radiographs in Fig. 10, a sound weld in all respects was made between Nos. 14 and 3. From Nos. 4 to 10, the weld was so made as to produce lack of penetration at the root of the weld and thus form a small crack at the root. A discontinuous dark line may be seen on the radiographs running centrally through the weld between Nos. 4 and 10. This is further substantiated by observation of the polished sections. A dark line of greater width is also seen in the radiographs running along the outer edge of the weld and between Nos. 7 and 9, and 11 and 13. This represents lack of penetration of the backing ring seal weld to the pipe and is completely without the main weld area as may be seen from polished sections 8-2, 9-1, 11-1, 12-1, etc. (Fig. 9).

The defect between Nos. 5 and 6 on the radiograph is that of a $\frac{5}{16}$-in. stove bolt and nut which was welded into the joint, the head of which is shown in the polished section No. 5-2. The dark streak near the extremity of the weld and opposite No. 6 on the radiograph is the result of a $\frac{3}{16}$-in. electrode stub approximately 2 in. long which was placed in the joint and covered over with weld metal. Considerable porosity is visible between Nos. 8 and 10 on the radiographs, some of which may also be observed from the corresponding polished sections. This condition of porosity was produced by welding over a heavy layer of slag from a previous pass. Further explanation of defects observed in these radiographs is believed to be unnecessary.

In addition to the study of weld defects in this experiment, a brief study was made on the effect of penetrameter location as it affects penetrameter image, about which A.S.M.E Boiler Code Case No. 953 deals.[3] In this experiment, two stepped penetrameters and two single-thickness penetrameters were used, one of each being placed on the source side and one of each being placed on the film side of the pipe. The images of all four penetrameters are visible in Fig. 10 (top).

As was expected, the images of the two penetrameters placed on the source side are somewhat enlarged.

From the standpoint of radiographic contrast, it seems that little if any difference should result regardless of which side of the material the penetrameter is located. From the standpoint of radiographic definition and sharpness of image, there is no question that a sharper penetrameter image will result if the penetrameter is placed on the film side in preference to the source side, though again, whether this difference in sharpness is noticeable to any appreciable extent will depend on both the source-to-film ratio and the size of the radium capsule (size of focal spot).

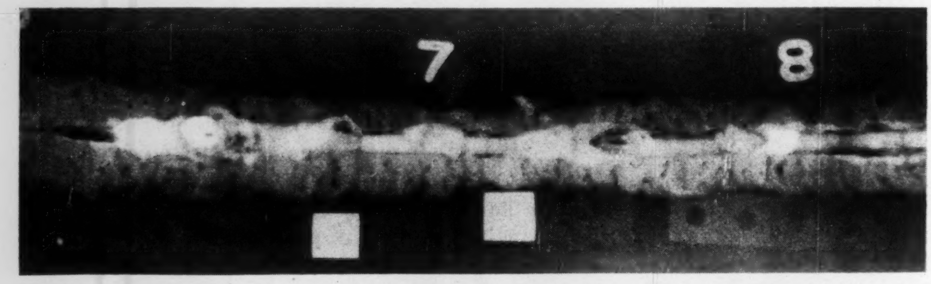


FIG. 11.—Gamma-Ray Radiograph Showing Pipe Weld Which Was Made with Bare Wire Approximately 15 yr. Ago.

It is the author's opinion that for all practical purposes little if any difference is to be seen in images of the penetrameters placed on both the source and film sides of the pipe as shown in Fig. 10 (top).

Figure 11 is a section of a $10\frac{3}{4}$-in. outside diameter $\frac{5}{16}$-in. wall pipe which was recently removed from an existing installation. This weld was made using bare wire and, as near as can be determined, placed in service about 15 yr. ago. A $\frac{3}{16}$-in. bare wire electrode used to fill in the root of the weld may be

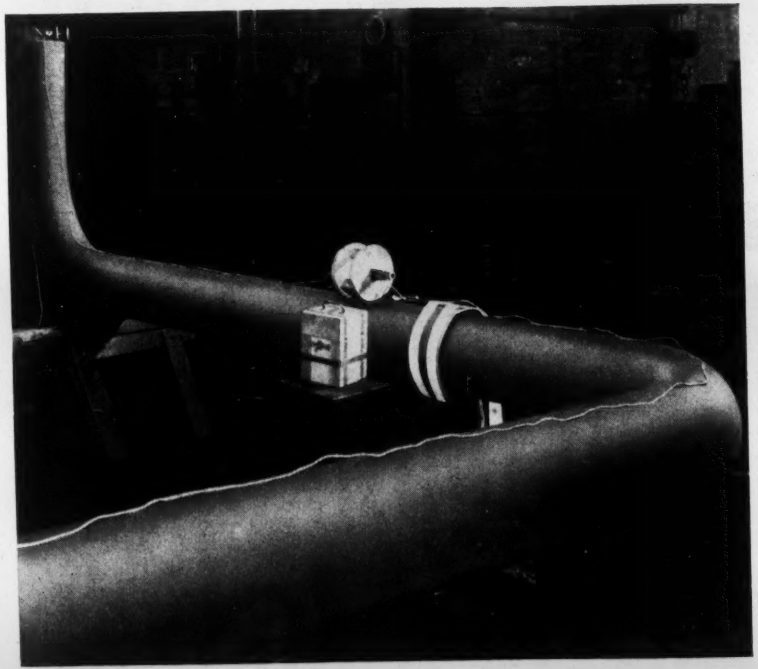


FIG. 12.—Setup Used for Taking Gamma-Ray Radiograph of Weld Between Two Pipe Bends.

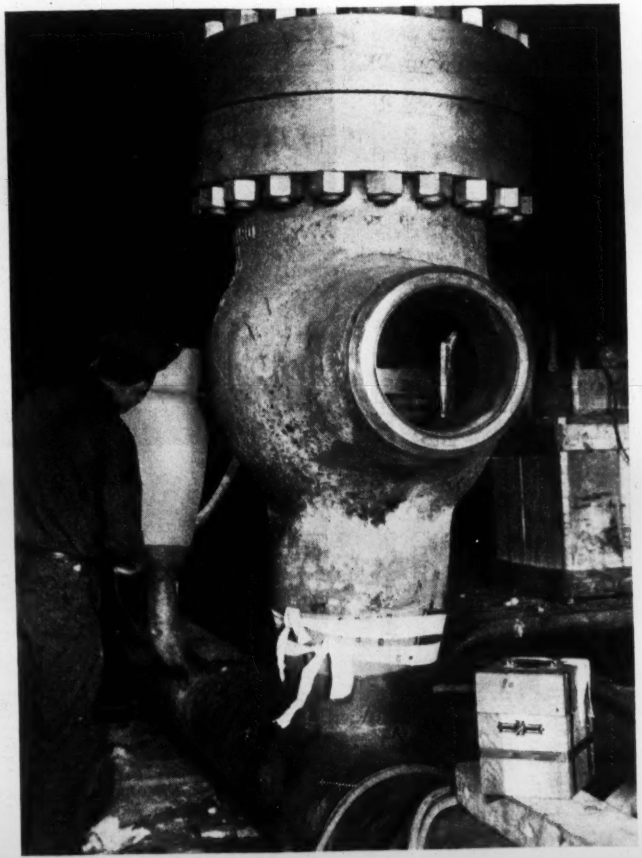


FIG. 13.—Radiographing a 14 in., 1500 psi. Nonreturn Valve to Boiler Superheater Header Outlet

seen in this radiograph; also lack of penetration, excessive porosity, "icicles," etc. The same penetrameter experiment was performed in connection with this radiograph and the results may be seen to closely parallel those of Fig. 10. The amazing point is, that such a weld actually did not fall apart.

APPLICATION TO COMPLEX WELDED PIPING ASSEMBLIES

Due to the complexity of pipe connections and piping systems in general, each job presents its own problems, several of which are illustrated. Figure 12 illustrates the procedure used in radiographing a weld in a $10\frac{3}{4}$-in. outside diameter 0.843-in. wall carbon-molybdenum steel double square pipe bend to be used in a main steam line which is to operate at 900 psi. and 900 F. The radium is placed within the end of the wooden carriage and pulled centrally through the pipe until opposite the weld, around which the film has already been placed.

Figure 13 illustrates the radiography of a welded joint connecting a 14-in., 1500 psi. nonreturn valve to a boiler superheater header outlet. From this photograph it may be seen that the valve stem could be raised only approximately 3 to 4 in. off the valve seat. In order to lower the radium centrally approximately 18 in. below the valve seat it was necessary to fasten the radium to the center of a wooden cross, the cross being suspended on fish line and supported by a board placed across the valve seat.

In contrast to this it is sometimes necessary to radiograph small diameter marine piping in which the radiograph must be taken entirely from without the pipe weld. Figure 14 shows a radiograph of a 4-in. standard pipe weld and backing ring taken through both walls of the pipe. This is accomplished by means of the procedure shown in Fig. 2 (e). In order to obtain the upper and lower quadrants which are shown to be underexposed it is necessary to rotate the pipe 90 deg. and make a second exposure.

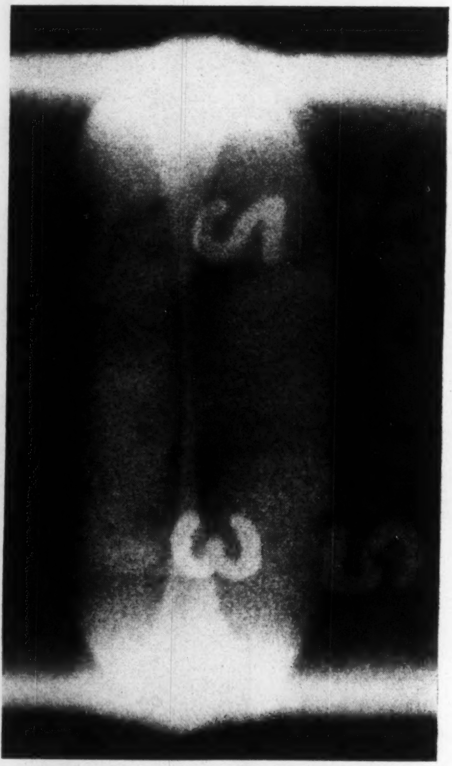


FIG. 14.—Gamma-Ray Radiograph of a 4-in. Pipe by Shooting Through Both Pipe Walls. Both radium and film external to pipe.

SUMMARY

Gamma-ray radiography is believed preferable to X-ray radiography for use in radiographing circumferential pipe welds, due to the relative simplicity and greater adaptability of the former method. This is particularly true where field radiography is required.

When radiographing circumferential pipe welds in which the radium is placed centrally, the source-to-film ratio seldom exceeds 7 to 1 and may be as low as 3 to 1. When working with such low ratios, the size of the radium source should never exceed 50 mg. and should preferably be 25 mg., if good radiographic detail is to be expected.

Under the most favorable radiographic conditions, the absolute sensitivity of the higher-speed coarse-grain films such as Eastman Type K, and Agfa Non-Screen, was found to be 0.02 in. The use of a slower-speed - fine-grain film, such as Eastman Type A, however, was found to have an absolute sensitivity of 0.01 in. or better. The type of film to be used will of course depend upon, first, the results desired and, second, the exposure time required.

From the results of test welds, as well as welds made in production, it is felt that the gamma-ray method of radiography will adequately pick up both major and minor weld defects.

Acknowledgments:

The author is grateful for the cooperation given by G. Sinding-Larsen, Chief Engineer; and G. W. Petrie, Jr., Plant Superintendent, Pittsburgh Piping and Equipment Co. The author also wishes to acknowledge the laboratory assistance given by Mathew Morrow.

Appreciation is expressed to the Pittsburgh Piping and Equipment Co. for their permission to publish the results of the various investigations performed.

REFERENCES

(1) R. F. Mehl, G. E. Doan, and C. S. Barrett, "Radiography by the Use of Gamma Rays," *Transactions*, Am. Soc. Steel Treating, Vol. 18, p. 1192, (1930).

(2) R. A. Gezelius and C. W. Briggs, "Radium for Industrial Radiography," Symposium on Radiography, published by American Society for Testing Materials (1943).

(3) G. E. Doan and Shang-Shoa Young, "Gamma-Ray Radiography," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part II, p. 292, (1938).

(4) J. T. Norton and A. Ziegler, "Sensitivity of the Gamma-Ray Method of Radiography," *Transactions*, Am. Soc. Steel Treating, Vol. 22, No. 3, p. 271, (1934).

(5) Norman L. Mochel, "Gamma-ray Radiography and Its Relation to X-ray Radiography," Symposium on Radiography and X-ray Diffraction Methods, Am. Soc. Testing Mats. (1936). (Symposium issued as separate publication.)

DISCUSSION

Mr. O. R. Carpenter.[1]—In discussing or comparing gamma rays with shadowgraphs, I think that perhaps we should not neglect the fact that not only do they produce shadows, but gamma rays also have the ability to penetrate and to be absorbed in steel, which does not make shadowgraphs as demonstrated by Mr. Emerson an exact analogy to the penetration of steel by gamma rays. From that standpoint I do not believe that the penumbra picture is exactly true.

The very fact about comparing a light shadow with a gamma-ray shadow is that light has absolutely no ability whatsoever to penetrate a defect, whereas your X-rays or gamma rays do have. In consequence, the image which you are getting on the film is not only the shadow of the outline of the defect, but it is the result of a difference in the amount of radiation reaching the film due to the differential in absorption of the beam through the defect as it is measured by the film. You not only have a shadow but you also have a differential in absorption recorded by the film itself.

The point I wished to bring out is that you have a certain degree of absorption by a beam of X-rays or gamma rays, which changes considerably the picture of film distance as worked out geometrically from the shadow standpoint. The very fact that the beam is absorbed does not make the geometric diagram work out exactly to the point.

Mr. R. W. Emerson.[2]—I am in complete agreement with Mr. Carpenter's remarks, realizing that due to the "depth effect," resulting from the differential absorption of gamma rays or X-rays by steel, quantitative measurements of image sharpness, penumbral shadow, etc., by the shadowgraph method would introduce error and that results obtained by this method would not be exactly comparable to radiographic results obtained under parallel conditions of d/t ratios, source size, etc.

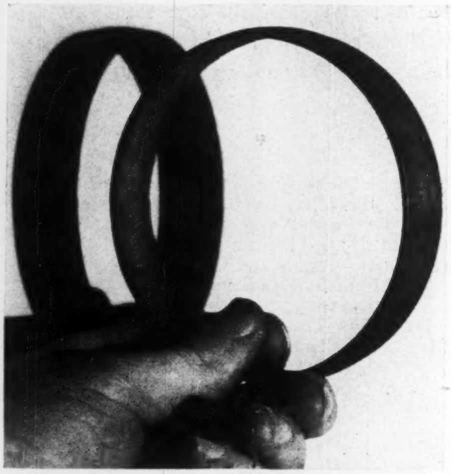


Fig. 1.—Light-Ray Shadowgraph Taken Under Conditions Comparable to Those of Fig. 14 of the Paper.

On the contrary, it was my intent that the shadowgraph analogy should be considered purely in a qualitative sense. From this viewpoint we may neglect the "depth effect" and consider the defect to lie in one plane.

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[1] Engineer, The Babcock & Wilcox Co., Barberton, Ohio

[2] Metallurgist, Pittsburgh Piping and Equipment Co., Pittsburgh, Pa.

I had only one purpose in mind, when showing the shadowgraphs of Fig. 4, namely, that whether we have ordinary light rays or gamma rays, a relatively large *d/t* ratio will produce a sharp shadow, while a small ratio will produce a shadow with indistinct or fuzzy edges.

To further illustrate that the general shape or contour of an ordinary light ray shadow is in a qualitative sense comparable to shadows produced by gamma rays, the accompanying Fig. 1 is given, to be compared with Fig. 14 of the paper.

The wide light area at the top and bottom of the pipe can be seen to taper to a narrow line at the center of the pipe in the radiograph of Fig. 14. This is due to differential absorption, about which Mr. Carpenter has spoken. This, of course, is not to be found in the accompanying Fig. 1, since the steel ring is opaque to white light.

I have, however, found the shadowgraph method very useful in explaining the general methods employed in gamma-ray radiography to persons who are supposed to interpret films but seem totally unfamiliar with the methods employed in the process.

MR. H. H. LESTER.[3]—Mr. Carpenter's point is well taken. The X-ray image is not a shadowgraph in the strict sense of the term because of the effect of the dimension of the defect in the direction of the radiation, which we may call a "depth" effect. However, if the size of the source of radiation, that is, the focal spot of the X-ray target or the size of the radium pill is very small, or the *d/t* ratio is sufficiently great, a sharp shadow will be obtained from any plane that can be drawn through the defect perpendicular to the direction of the radiation. This condition will be met if sharply defined images of the holes of the Boiler Code type of penetrameter are registered on the negative.

It has been our experience that much larger working distances are required than those indicated by Mr. Emerson, if sharp images are to be obtained. It is true that there will be a registry of defects for short working distances where the *d/t* ratio is less than that required for sharp images. That is, there will be relative contrasts that are indicators of the presence of defects. But such images are not always easily interpreted, may be misleading, and images of fine cracks may be missed altogether.

It is believed that the present trend in radiography is toward better images. The advocacy of lower *d/t* ratios than will give good images is not in line with this trend.

MR. C. A. ADAMS.[4]—I should like to ask whether Mr. Emerson has come to any conclusion as to the minimum size of penetrameter hole which will show up clearly on the films with a distance ratio as low as three.

MR. EMERSON.—I have no exact information available using a ratio as low as three. The 14-in. outside diameter by 1.4-in. wall pipe discussed in my paper, produced a *d/t* ratio of four. A standard type Boiler Code penetrameter was used which was originally made for a steel thickness of 1.3 in.

The penetrameter thickness was 0.026 in. and the diameter of the holes was 0.062, 0.078, and 0.104 in., respectively. All three holes were actually visible when the penetrameter was placed on either the film side or the source side, with definition sufficient so that you could easily pick the holes out. Naturally you are going to get a slight enlargement of the penetrameter image on the source side, but the holes actually showed up so that there was no question of the fact that the holes were present. The exposures were made with a 25-mg. capsule. The diameter of the source was about $\frac{1}{16}$ in.

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[3] Principal Physicist, Watertown Arsenal, Watertown, Mass.

[4] Professor of Engineering, Emeritus, Harvard University, Cambridge, Mass.; and Consulting Engineer, Edward G. Budd Manufacturing Co., Philadelphia, Pa.

MR. LESTER.—That is the point I tried to make a while ago. You can see those penetrameter images because you have difference of contrast, but you will not see them with sharp edges unless you get rid of the penumbral shadow, which destroys detail and is objectionable for some types of work. With the smaller capsule, you can use a smaller ratio. With a 200-mg. pill, the chances are you will have to have a *d/t* ratio of around 20.

MR. ALEXANDER GOBUS.[5]—In connection with our power plant piping, and the inspection thereof with radium, we have used for a number of years nearly the same method that Mr. Emerson has described, with the exception that we have used single lengths of film. We find it of great advantage to buy 5 in. wide film 50 ft. long, take it out in the field, and cut it to suit the job. When we hand the film to an inspector he has just one picture to look at, and does not need to piece together a number of separate films from the same circumferential weld.

We have worked this film up to 50-in. diameter shells. However, we do not use a single film for that large diameter. We use up to 5 ft. successfully. We have devised a stainless steel fixture to put the film on, coil it up in a reel and drop it in a portable tank out in the field. In using this film in the field, there are very few people who have a satisfactory dark room, especially in the smaller plants or around a power plant, but so far we have had considerable success in being allowed to work in the local hospitals, and every one of them has had a bench about 6 ft. long that we could use. We carry around as our portable tanks enameled pots (like the steamer pot in a kitchen), about 12 in. in diameter by 14 in. high, and we use our own developer and hypo in the hospitals.

Instead of carrying powders and going through all the trouble of mixing them, we use the concentrated liquid developer and concentrated liquid hypo. It costs only a few cents more, but saves a lot of trouble in the field.

MR. GILBERT E. DOAN.[6]—Coming back to this question of fuzziness and focal distance, as long as the source is larger than the flaw you are going to get some haze. It is true that the amount of haze will diminish as you increase the distance, but you will always get some, and I do believe that the distances that are being used are in many instances much longer than necessary.

We reported on a series of experiments a few years ago in which we made definite flaws of definite width and depth in blocks of steel of different thicknesses, and reported detection of those flaws with much shorter distances than are commonly used.

It is true that you do not get the same degree of definition, but in many instances you can put up with a good deal of haze if it will permit you to shorten your focal distance and exposure time, and I have a very definite conviction that some of the distances that are being used can be cut down in instances where you do not need that high degree of sensitivity.

THE CO-CHAIRMAN (*Mr. James J. Curran*[7]).—Mr. Doan's remarks are very much to the point. In some of our own practice on castings we elect to use short distances to speed up exposure so that we can cover a casting 100 per cent. We know that we will not get the optimum definition or sensitivity, but we feel that we will get some sign of the defect if it is present, and we can always make a single shot under better conditions to bring that out more definitely.

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[5] Lucius Pitkin, Inc., New York, N. Y.

[6] Professor of Metallurgy, Lehigh University, Bethlehem, Pa.

[7] Chief Metallurgist, Walworth Co., Inc., Greensburg, Pa.

MR. ADAMS.—Are these fine-grained films, which I understand are directly sensitive to the radiation without transmission through a screen, equally sensitive to ordinary light radiations?

THE CO-CHAIRMAN (*Mr. H. E. Seemann*[8]).—No, they are not. They are ordinarily considerably slower to light than the screen films. In making up that type of film no attention need be paid to that particular factor.

MR. ADAMS.—In other words, they are easier to handle in light?

MR. SEEMANN.—Yes. They are safer in the darkroom. If a person is using one type of film right along, there is always a possibility of his being able to increase the amount of light in his dark room, provided he makes satisfactory safelight tests himself.

MR. CURRAN.—This subject of the sensitivity of films is one that has worried us at times. Some time ago in our own plant a copying camera was installed to copy tracings and so forth for the record. When we became aware of the fact that this apparatus had been placed in a specially constructed room right next to our radium room, we sounded the alarm immediately. Everybody was very much afraid of what would happen to the film that was already stored there, but we made some tests and were rather amazed to find that the Recordak film could be exposed in a sealed envelope for some time, we think it was actually 24 hr., within a few inches of a 300-mg. capsule of radium without any fogging. So all film is not equally fogged by radium.

MR. SEEMANN.—We do not know of any photographic emulsions that are not sensitive to gamma radiation, but they vary in sensitivity between wide limits. I think if you had been willing to wait long enough you could have obtained some exposure on the Recordak film.

MR. GOBUS.—About two years ago I was caught without film, without likelihood of getting about 3 ft. of film for three or four days, and I used a roll of Verichrome, took a pot shot, gave it double exposure, and had a satisfactory film.

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[8] Physicist, Eastman Kodak Research Laboratories, Eastman Kodak Co., Rochester, N. Y.

AN INVESTIGATION OF THE APPARATUS USED IN RADIUM RADIOGRAPHY

By L. W. Ball[1] and D. R. Draper[2]

In radium radiography, the apparatus used is simple compared with X-ray apparatus, and secondary radiation is relatively unimportant. However, there is still a considerable difference between the good quality of radiographs obtained with a suitable choice of apparatus carefully arranged and the poor quality that can result from unsuitable apparatus and careless arrangement.

The object of the experiments described in this paper was to provide guidance to radiographers in the selection and setting up of their apparatus by measuring the degree to which variations in each feature of the apparatus affect the radiographic sensitivity.

These features will be considered under four headings. They are: (1) the source, consisting of the active substance and the capsule in which it is sealed; (2) the surrounding material consisting of the walls of the room, table top, etc., together with the protective housing used to limit the spread of the radiation; (3) the cassette, containing the film and screens; and (4) the photographic equipment consisting of the film, processing solutions, and viewing lamps.

The use that the radiographer can make of the results of these experiments will depend very much on his subject. For thin material containing fine flaws remote from the film, the effect of the source size is most important, while for heavy castings containing only coarse cavities, the effect of thick lead screens and of the protective housing in reducing external secondary radiation is most important. Fortunately, however, the features that are especially important in certain cases are quite desirable in general practice and a complicated variety of techniques is not necessary.

The Source

Table I presents the specifications of three different gamma-ray sources. Any difference in the quality of the radiographs that they produce is due solely to differences in their effective size. In some radiographic problems (see Fig. 1 (a)), a small source is the only way to

TABLE I.—SPECIFICATION OF GAMMA-RAY SOURCES.

Type	Shape	Diameter	Length, mm.	Content[a]	Capsule Wall
A	sphere	1 mm. (0.04 in.)	1	100 mc. radon	1 mm. glass
B	cylinder	3 mm. (0.12 in.)	3	30 mg. radium	1 mm. silver
C	cylinder	6 mm. (0.24 in.)	6	250 mg. radium	1 mm. silver

[a] Normal radon gas is not highly concentrated. If necessary, 1000 mc. could be enclosed in the 1-mm. bulb. Further concentration of the radium salt would be prohibitively expensive.

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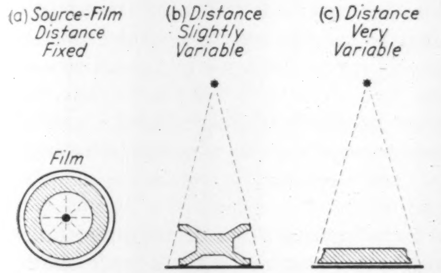
[1] Research Physicist, Triplett & Barton, Inc., Burbank, Calif. Formerly with the National Research Council of Canada.

[2] Radiographer, Inspection Board, United Kingdom and Canada, Ottawa, Ont.


(a) Source-Film Distance Fixed
(b) Distance Slightly Variable
(c) Distance Very Variable
Film

(Cast bomb case) (Tank wheel) (Cast armor plate)

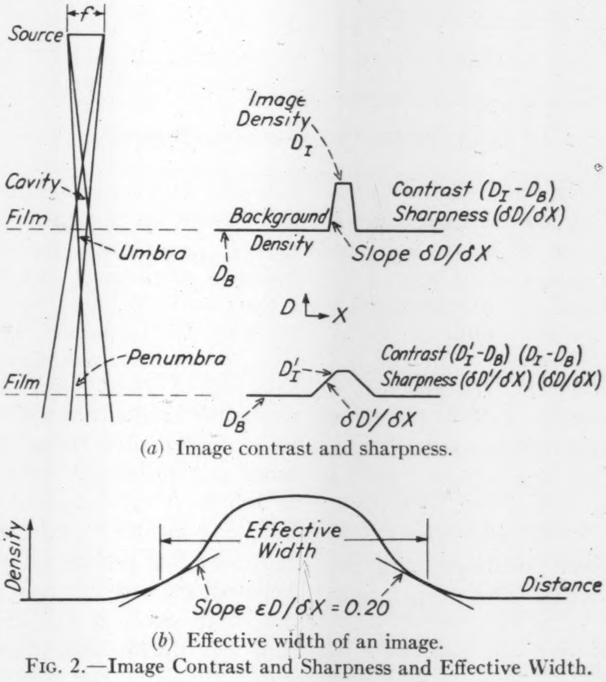
FIG. 1.—Representative Gamma-Ray Subjects.

get very sharp images of narrow cavities, but in general, the detrimental effect of a large source size (f) can be compensated by using a large source-film distance (S. F. D.).

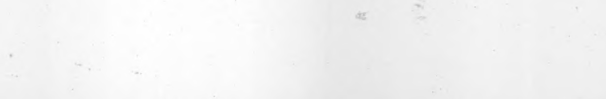
To make this compensation some simple formula is needed to give a relation between S. F. D. and f that will provide the same degree of image legibility for narrow cavities.

A radiographic image of a cavity of constant depth consists of a background of uniform film density D_B, a penumbra region where the density is changing at an average rate of $\delta D/\delta X$ and a central umbra or penumbra region of constant density D_I (Fig. 2). The legibility of the image depends first on the total contrast $(D_I—D_B)$ which must exceed 0.02 density units, and second on the sharpness $\delta D/\delta X$ which must exceed about 0.2 density unit per inch.

If the contrast and sharpness exceed the threshold values, the human eye can detect the image of a cavity no matter whether it is an umbra or a penumbra image. But for a cavity of a given depth the contrast of the image decreases when the umbra is lost; therefore, a suitable formula for determining the source-film distance should ensure


f
Source
Cavity
Film
Umbra
Penumbra
Film
Image Density D_I
Background Density
D_B
Slope $\delta D/\delta X$
Contrast $(D_I - D_B)$
Sharpness $(\delta D/\delta X)$
D
X
D_I'
D_B'
$\delta D'/\delta X$
Contrast $(D_I' - D_B)$ $(D_I - D_B)$
Sharpness $(\delta D'/\delta X)$ $(\delta D/\delta X)$

(a) Image contrast and sharpness.


Density
Effective Width
Distance
Slope $\varepsilon D/\delta X = 0.20$

(b) Effective width of an image.

FIG. 2.—Image Contrast and Sharpness and Effective Width.

that the smallest cavity diameter that casts an umbra shadow is the same with each source. This cavity diameter may be called the "umbra limit" and it is equal to the source diameter divided by the ratio of the source-film distance to the cavity-film distance, that is, the umbra limit $U = f \div (\text{S. F. D.}:d)$.

For example, with S.F.D.:d = 4:1 the umbra limit for a 250-mg., 6-mm. diameter radium source is $1\frac{1}{2}$ mm., but for a 250-mc., 1-mm. diameter radon source, the umbra limit is only $\frac{1}{4}$ mm. Alternately, to obtain an umbra limit of $\frac{1}{4}$ mm. with a cavity-film distance of 3 in., the radon should be used with a source-film distance of 12 in. and the radium with a distance of 72 in.

Doan and Young (1)[3] have preferred a formula based on an umbra $\frac{1}{32}$ in. in diameter and an allowance for the obliquity of the radiation. However, it is not possible to obtain exactly the same sort of image with different source sizes, so elaboration of the simple "umbra limit" criterion is not an advantage.

So far, the "effective source" of radiation has been identified with the radioactive material. However, it cannot be assumed that the theoretical shadows cast by the radioactive part of the source are the only factors in revealing fine defects. Secondary radiation from the radiographic subject modifies the image in quite a complicated way (2) and secondary radiation is also emitted by the capsule.

Experimental tests of the importance of these secondary effects were made. Figure 3 shows the arrangement used to study the effect of scatter from the capsule.

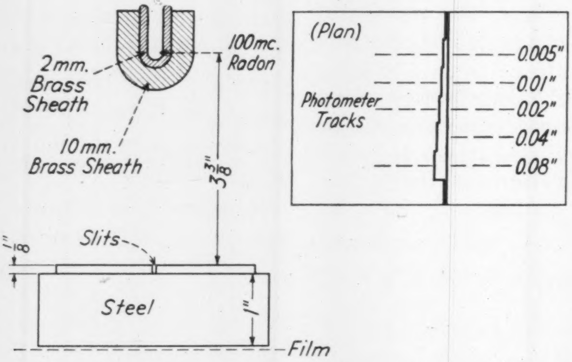


FIG. 3.—Scatter from Capsule and Holder.

The source consisted of a glass capillary tube with a 1-mm. internal diameter bulb blown on the end and containing radon. In practice, radium is usually sealed in small silver capsules with 1 or 2-mm. wall thickness and these small capsules are contained inside a brass, steel, or aluminum holder with a wall thickness of about 1 cm. In the experiments the smaller brass sheath was used to represent silver capsules and larger brass and aluminum sheaths were used to represent typical holders. The slits in the $\frac{1}{8}$-in. steel plate were used to represent fine defects. Four films were exposed, one with the glass tube only and one with each of the sheaths. Each film was microphotometered along the paths shown in Fig. 3. Neither in the photometer curves nor in visual examination of the films could any differences be found that were due to the presence of the sheaths.

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[3] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 1143.

Therefore, in considering the design of a radium capsule or holder, secondary radiation produced by them may be neglected.

To study the importance of the internal diameter of the capsule, the three sources described in Table I were used. To compare these sources experiments similar to those illustrated by Fig. 3 were made, but the details were arranged to distinguish the following conditions. When a defect is so near to the film that all the sources cast umbra shadows, each source should give the same contrast, that is, the same difference in density between the center of the image and the background. Under this condition only very minor differences in legibility due to different density gradients at the edge of the image may be expected. When a defect is at such a distance from the film that it casts an umbra shadow with a small source, but only a penumbra shadow with a large source, marked differences in both the contrast and the sharpness of the images will be expected. In either case, when conditions are such that secondary radiation from the steel block contributes greatly to the image, this contribution will be independent of small differences in the source size, and so it may make the primary differences unimportant.

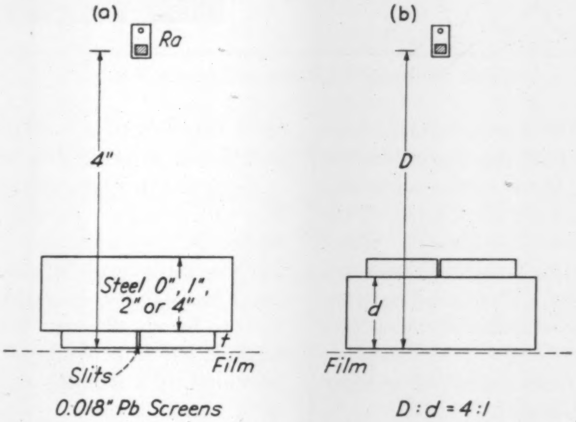


FIG. 4.—Umbra and Penumbra Images (Primary Radiation).

One series of experiments was carried out with the arrangement shown in Fig. 4(*a*). In these exposures primary radiation always cast an umbra shadow of each slit and secondary radiation had the maximum chance to contribute to the images. Under these conditions differences between the three sources should be reduced to a minimum. Photometer curves were made and they, together with visual comparison of the films, showed that the effect of source size under these conditions was quite negligible.

It follows that in the radiography of cast or welded plates, or other materials placed very close to the film, a small source is not essential.

A second series of experiments was carried out with the arrangement shown in Fig. 4(*b*). In these exposures primary

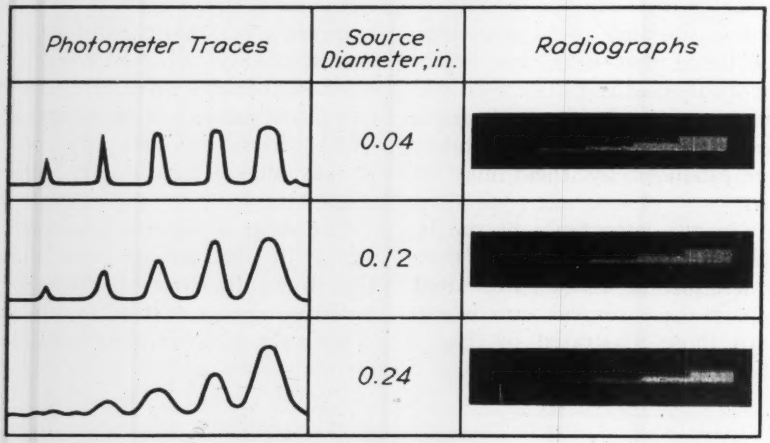


FIG. 5.—Image Legibility and Source Size.

radiation from the 1-mm. source cast umbra shadows of all the slits, from the 3-mm. source of three of the slits, and from the 6-mm. source of only two of the slits. Under these conditions, which produce some umbra images without any scatter assistance, differences between the three sources should be most pronounced.

Figure 5 illustrates the striking differences actually obtained.

Results of this nature are not very useful to the practical radiographer unless some lucid method of generalization is introduced. Table II is an attempt to express the present data in a manner that is applicable to practice.

In compiling this table a value of $D:d = 4$ was used with source diameters of 0.04, 0.12, and 0.24 in., but the results can be applied to any other combinations giving the same umbra limits.

TABLE II.—RELATION OF CRACK SENSITIVITY TO UMBRA LIMIT.

Steel block 1.25 in. thick.

Umbra limit $\frac{fd}{\text{S.F.D.}}$, in.	Minimum Depth of Visible Slit, per cent ($\partial D = 0.03$)				
	0.01-in. Slit Width	0.02-in. Slit Width	0.04-in. Slit Width	0.06-in. Slit Width	0.08-in. Slit Width
0.01	8	5	5	5	5
0.03	15	9	5	5	5
0.06	30	15	8	6	5

To apply this type of table to practice, the radiographer should calculate the umbra limit for a proposed inspection and use the table to determine whether the crack sensitivity is satisfactory. If it is not, he should use the table to determine the improvement that would be provided by a smaller umbra limit, that is, by a smaller source or larger $D:d$ ratio.

The two experimental arrangements already described represent normal radiographic conditions. Cases where scattered radiation might assist in forming images of cavities remote from the film do not occur in testing welds or castings of ordinary thickness. However, there remains the case of testing very thick plates by placing radium in contact with one side and a film in contact with the other. The question arises: is the size of the source important in this case, or does the image-producing effect of secondary radiation mask primary differences? An experimental test of this case was made using a composite block of steel 6 in. thick. Slits in a $\frac{1}{2}$-in. plate were included in the block between $1\frac{1}{2}$ and 2 in. from the film surface. The resulting films showed that the dependence of image legibility on source size was not decreased very much by the contribution of secondary radiation from the 4 in. of steel above the slits.

The net results of the four groups of experiments just described are that in any practical consideration of the effect of source size, secondary effects can be neglected and that the relative legibility of narrow images can be anticipated by calculating the simple umbra limit and applying experimental data in the form shown in Table II.

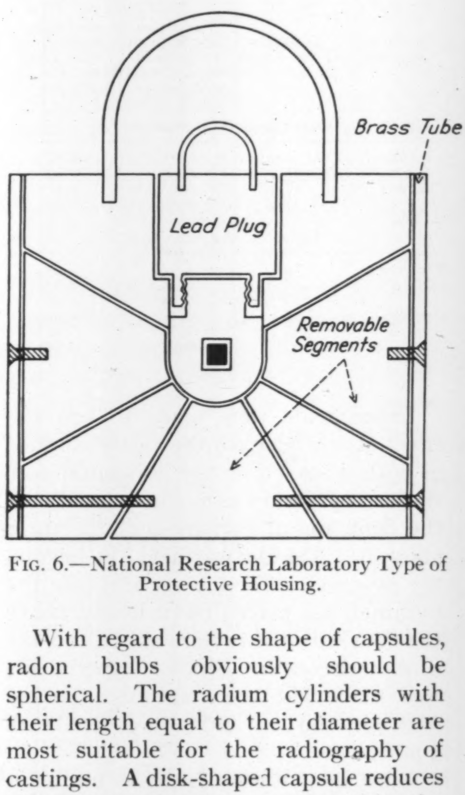


FIG. 6.—National Research Laboratory Type of Protective Housing.

With regard to the shape of capsules, radon bulbs obviously should be spherical. The radium cylinders with their length equal to their diameter are most suitable for the radiography of castings. A disk-shaped capsule reduces absorption of gamma rays within the radium and facilitates stacking of several units, but the larger source diameter is only justified when the $D:d$ ratio has to be large because of considerations other than the umbra limit, as for example in the radiography of flat plates.

If a source much more powerful than 250 mg. is required for use with a $D:d$ ratio less than 4:1, the only way to obtain an umbra limit less than $\frac{1}{16}$ in. is to use radon. When the geometry of the object enforces a $D:d$ ratio of less than 2:1 and a source more powerful than 30 mg. must be used, radon is again the solution. Gun barrels or breaches radiographed from the inside are the most obvious examples of need for radon.

THE SURROUNDINGS

In radium radiography gamma rays are emitted in all directions. In a room radiation strikes all the walls and other objects, and produces scattered radiation from them. At any point on the film there may be intense local scatter from some nearby object, but in addition there will be a general scatter intensity built up by the whole enclosure. To reduce this intensity, either the walls and tables may be covered with material that scatters little, or they may be removed; that is, radiography may be performed outdoors. As will be proved later, the worst place to use gamma radiography is in a small brick or concrete enclosure, but when such conditions are unavoidable, considerable improvement can be obtained by using a protective lead housing close to the source.

Figure 6 shows the National Research Laboratory type of protective housing. Because scatter from such a housing can often undercut the edges of castings, experiments were made to measure its intensity. A steel block 4 in. thick with slots in its upper surface was radiographed with the radium vertically over one edge. Exposures were made with a capsule only; with a series of brass, steel, and aluminum holders; and with the lead housings. In each case the intensity of the undercutting secondary radiation was quite negligible.

To study the factors that effect general room scatter, a special series of experiments was devised which may be called the "scatter-box" experiments. A lead pyramid and boxes of iron, lead, brick, and wood were made and set up as shown in Fig. 7. The 4-in. lead pyramid reduced the intensity of the direct radiation reaching a film either in position *F* or *B* by a factor of 250.

In a preliminary experiment, the lead pyramid was fixed on the laboratory roof with the film on top (position *B*) and the radium in position underneath. With this arrangement, only direct radiation could reach the film because the scatter from the sky was negligible. It was found that the direct radiation produced a density of 1.0 (above the background density of 0.15) on direct-exposure film between 0.018-in. lead screen in 5.8 hr. When the same pyramid and radium source were inserted in the "scatter boxes" a film blackening of 1.0 was obtained in 1.35 hr. with 4-in. thick brick walls, in 2.3 hr. with 4-in. thick wood walls, in 3.75 hr. with $\frac{1}{8}$-in. thick lead walls, and in 2.0 hr. with $\frac{1}{2}$-in. thick iron walls.

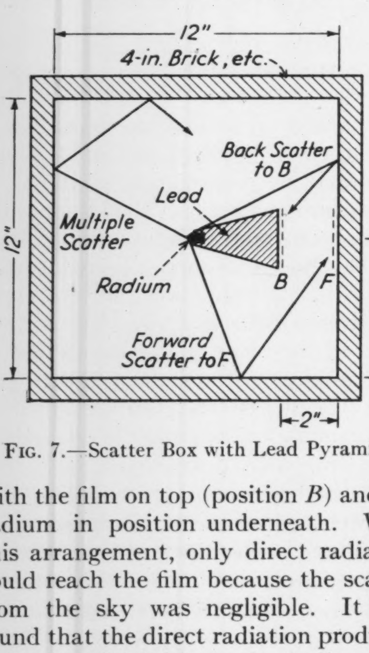


FIG. 7.—Scatter Box with Lead Pyramid.

In practice, the radium is not placed in mid-air equidistant from six similar walls (floor and ceiling included), but it is placed fairly close to the floor or to a table top. It is important to know whether this close surface is the predominant source of scatter.

The radium, casting, and film are either set up in a horizontal arrangement along the floor or table top, or they are set up in a vertical position with the radium suspended above the film which lies on the floor or table.

TABLE III.—SCATTER FROM NEAREST SURFACE.

Material	Distance, Film Above Surface, in.	Blackening Rate Without ⅛-in. Lead Sheet, density units per hr.	Blackening Rate With ⅛-in. Lead Sheet, density units per hr.
HORIZONTAL ARRANGEMENT			
2 in. wood....	1¼	0.25	0.09
	13¼	0.06	0.03
Concrete......	1¼	0.60	0.17
	13¼	0.16	0.11
VERTICAL ARRANGEMENT			
2 in. wood....	0	0.14	0.03
	2	0.09	0.03
	12	0.04	0.03
Concrete......	0	0.12	0.02
	2	0.10	0.02
	12	0.07	0.02

Experiments were made to test the effect of scatter from the closest surface in both cases. For the horizontal case the lead pyramid was mounted parallel to the floor and at various small distances above it. The film was held 2 in. beyond the pyramid. For the vertical case the pyramid was placed immediately above the floor with the radium above and the film underneath. Similar experiments were made with the wood table top and with both the floor and table covered with $\frac{1}{8}$ in. of lead.

Once again it is necessary to find some lucid way of expressing the experimental measurements in a form in which they can be applied to practice. The best way to do this is to state the film blackening rate. For example from the scatter-box experiment, it can be stated that scatter produced a blackening of 0.6 density units per hour from brick, 0.3 density units per hour from steel, 0.15 from wood, and 0.07 from lead.

The results of the "close surface" experiments are given in Table III.

The intensity of the wall scatter will depend greatly on the size of enclosure. A very small room will be worse for radiography than a large one. To obtain some measurements of this effect, several sizes of brick scatter boxes were tested. A box 2 by 2 by 2 ft. produced 0.3 density units per hour, and a room 25 by 25 by 18 ft. produced only 0.02. The actual values of the scatter blackening rates quoted above apply only to the local conditions under which they were made. However, the lead pyramid arrangement that was used for them is very convenient for testing the local intensity of scatter in any field inspection.

THE CASSETTE

The problem of designing a suitable cassette for radium radiography is largely a matter of deciding the thickness of the lead screens. Ordinary fluorescent screens can be used only for low-sensitivity work because they are much more sensitive to secondary radiation than to primary gamma rays and their filtering effect is quite inadequate.

Experiments were made to test the dependence on screen thickness of the intensification of the image, the sharpness of the image, and the reduction in forward and back scatter.

In order to separate the first and third of these factors, the lead pyramid was set up on the laboratory roof with a film in position *B* and facing the sky. Film were exposed with a back screen made of a strip of cardboard in contact with one part of the film and strips of antimony lead foil in contact with other parts. The densities obtained showed that 0.009-in. lead back screen is thick enough to give full intensification. Similar experiments showed that a 0.009-in. lead front screen also was thick enough to give full intensification.

To study the dependence of image definition on lead screen thickness, a plate containing fine slits was radiographed with films between pairs of lead screens 0.003, 0.006, 0.012, 0.021, and 0.40-in. thick. These films were examined visually and also microphotometered. No decrease in image sharpness with screen thickness was found for the film that was used.

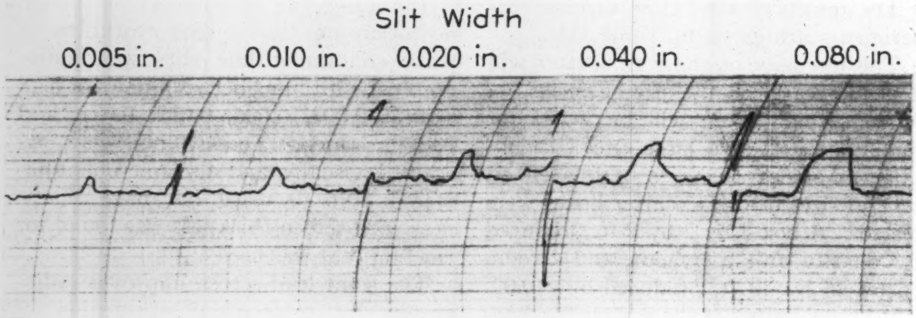
The front lead screen improves radiographs by preferential absorption of forward scattered radiation, some of which is considerably softer than the primary gamma rays and some of which has to pass through the screen at an obtuse angle. The back screen absorbs some of the back scatter from the walls. In both cases, it is desirable to increase the absorption by using very thick lead screens. An upper limit to the thickness of each lead sheet of about 0.020 in. is necessary to give the flexibility required to maintain close contact between the screen and the film, but thicker screens may be made of several layers. Also very thick screens would allow an undercutting by secondary radiation from the lead at the edges of the image of thick subjects in the manner fully demonstrated for 200-kv. X-rays by Seemann (3).

TABLE IV.—ABSORPTION OF BRICK SCATTER BY LEAD SCREENS.

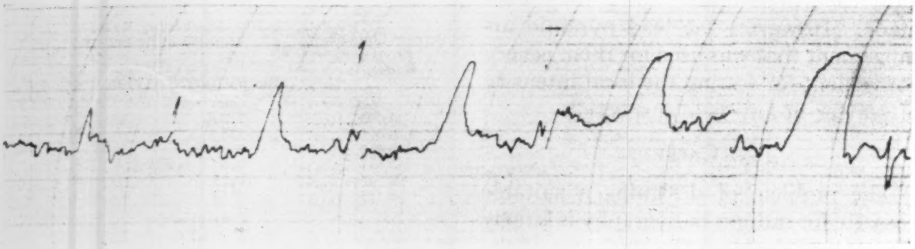
Screen Thickness (Front and Back), in.	Blackening Rate, density units per hr.	
	Film position *B*	Film position *F*
0.003	1.8	3.1
0.005	1.5	2.8
0.010	1.0	2.1
0.020	0.6	1.3
0.040	0.25	0.8
0.125	0.10	0.3

The small brick scatter box provided a

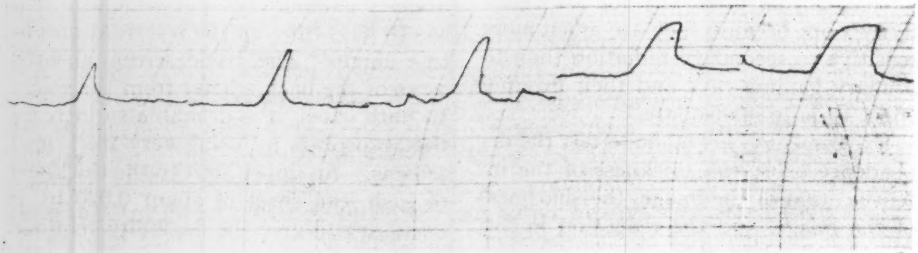
good way of measuring the efficiency of different thicknesses of lead screen. Table IV presents some interesting results. A compromise screen thickness of 0.040 in. has proved to be suitable for general use with thick castings. Under conditions where scatter absorption is relatively unimportant, thinner screens are more generally used because of their cheapness and flexibility.



Screen film, medium grain.



Direct film, coarse grain.



Direct film, fine grain.

FIG. 8.—Sensitivity and Grain Size.

When no part of the intense direct beam passes through the cassette, scatter from the cassette material is unimportant. When this is the case and the exposures are very long, it is good practice to reduce back scatter by adding a heavy ($\frac{1}{4}$ to 1-in.) lead screen immediately behind the cassette.

PHOTOGRAPHIC EQUIPMENT

In radium radiography with lead intensifying screens, certain readily available direct-exposure films give the greatest available speed, contrast, and radiographic range. The only disadvantage of these films is the rather large grain size which is visible when viewing the radiographs.

In radium radiography of castings the lateral size of most of the cavities is too great for the film grain size to have any marked effect, but in the examination of welds and forgings some defects give images that are considerably more legible on fine-grain film. Figure 8. presents three photometer traces made with a very fast film developed in Kodalk[4] and with an ordinary film and a fine-grain film developed in standard developer. The oscillations in background density and the obvious superiority of fine-grained film for narrow images show that the grain size differences are not eliminated by the spread of the intensifying electrons from the lead screens. Both photometry and visual inspection show only a slight disadvantage in the use of fast coarse-grained film for registering images widths of 0.040 in. or more.

In radium radiography it is desirable to make the most of the gain in speed obtained with a long developing time and the gain in contrast obtained with high film density. For both these purposes agitation of the films or circulation of the developing solution is an advantage. For the latter purpose the need for high intensity illumination already has been stressed by several authors (**4**, **5**).

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[4] The authors were under the mistaken impression that the choice of developer was important. A report of extensive experimental work from the laboratories of Triplett & Barton, Inc., included results demonstrating that the grain size was not much affected by the type of developer used.

REFERENCES

(1) G. E. Doan and Shang-Shoa Young, "Gamma-Ray Radiography," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part II, p. 292 (1938).

(2) G. E. Doan, "Gamma-Ray Radiographic Testing," *Journal*, Franklin Inst., Vol. 216, p. 183 (1933).

(3) H. E. Seemann, "The Reduction of Secondary Radiation and of Excessive Contrast by Filtration," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 1289 (1940).

(4) H. E. Seemann, "Trends in the Technique of Industrial Radiography," ASTM BULLETIN, No. 115, March, 1942, p. 21.

(5) L. W. Ball, "High Sensitivity in Radium Radiography of Castings," ASTM BULLETIN, No. 116, May, 1942, p. 29.

DISCUSSION

Mr. C. A. Adams.[1]—Is the unit density mentioned in the paper a normal density?

Mr. L. W. Ball.[2]—In radium work we usually aim at a density of about 2.0 to 3.5. The blackening rate, expressed in density units per hour, is used to calculate the exposure.

Mr. Don M. McCutcheon.[3]—The author mentioned the intensification with about 0.009 in. of lead. From what thickness of lead do you think the intensification of the electrons would come?

Mr. Ball.—We have measured the intensification produced by various thicknesses of lead. If the speed with no lead screen is unity, with 0.001 in. of lead our speed goes up to 1.6. With 0.002 in. of lead our speed goes up to 1.9; with 0.003 in. it is up to about 2.1, and when you get up to 0.009 in. the speed is 2.5, and from then on there is no increase. If you have lead 1 ft. thick, the speed will still be 2.5.

I mentioned the description of electron intensification in a paper by Mr. Doan.[4] The limiting of intensification to the surface layer is not, of course, because the gamma rays do not get into the lead; it is because the electrons cannot get out. The same principle applies to scatter.

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[1] Professor of Engineering, Emeritus, Harvard University, Cambridge, Mass.; and Consulting Engineer, Edward G. Budd Manufacturing Co., Philadelphia, Pa.

[2] Research Physicist, Triplett & Barton, Inc., Burbank, Calif. Formerly with the National Research Council of Canada.

[3] Metallurgical Engineer, Main Laboratory, Rouge Plant, Ford Motor Co., Dearborn, Mich.

[4] G. E. Doan, "Gamma-Ray Radiographic Testing," *Journal*, Franklin Inst., Vol. 216, p. 183 (1933).

AN EXPOSURE METER FOR X-RAY RADIOGRAPHY[1]

By Herbert Friedman[2] and Arthur L. Christenson[2]

Synopsis

An X-ray exposure meter has been developed for use with a 220-kv. X-ray radiographic installation at the Naval Research Laboratory. For given brands of film and intensifying screens, the meter may be calibrated to indicate the exposure times required for any desired film blackening. The dependence of this calibration on kilovoltage, specimen material, and filter technique has been investigated. In routine radiography, it is possible to obtain satisfactory results with a single calibration curve, at all voltages between 125-kv. and 200-kv., for any specimen material, and for filtered as well as unfiltered radiation. The receiving element of the exposure meter is a Geiger-Muller counter designed to permit accurate intensity determinations in a few seconds, with the measured beam of radiation limited to a pencil of X-rays, ¼ in. in diameter. The use of a small aperture on the counter permits the scanning of portions of castings of nonuniform thickness to determine an optimum kilovoltage and suitable filter technique for radiographing. Since the counter is placed close to the film position, the meter indications are independent of variations in the source-to-film distance.

It is essential for the proper interpretation of radiographs that the photographic density fall within certain limits. The general practice among radiographers is to determine exposure times from standard technique charts for X-ray radiography and from the gamma-ray slide rule when using radium. A given technique chart is applicable only to a single source-to-film distance, one type of specimen material, and a particular filter thickness. For every variation in these three factors, a new technique chart is required. The great dependence of X-ray absorption on the voltage applied to the tube and on the thickness, density, and atomic number of the specimen material, makes it difficult for the radiographer to select proper exposures with the meager data ordinarily available from technique charts. An X-ray exposure meter has therefore been designed which enables the radiographer to determine the correct exposure almost independently of these variables.

Design of Exposure Meter

A Geiger-Muller counter of the type shown in Fig. 1 is the sensitive element of the X-ray exposure meter. The common form of G-M counter is simply a cylindrical cathode and a centered anode wire, enclosed in a glass envelope. Normally, the tube is filled with a fraction of an atmosphere of a suitable gas, or mixture of gases, and a potential difference of the order of 1000 v. is applied to the electrodes. To operate the tube as a

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[1] Published by permission of the Navy Dept.

[2] Associate Physicist, and Junior Metallurgist, respectively, Division of Physical Metallurgy, Naval Research Laboratory, Anacostia Station, Washington, D. C.

G-M counter, the voltage is set to a value somewhat less than is necessary to maintain a self-sustained discharge between the electrodes. All that is then required to trigger a momentary discharge is the passage of some ionizing radiation through the counter. By proper construction of the counter and its associated circuit, a discharge triggered by the absorption of a quantum of radiation may be quenched very quickly. To determine the intensity of radiation entering the counter, one measures the number of discharge pulses produced per unit time.

In designing an exposure meter for X-ray radiography, it was desired that the meter have a period of only a small fraction of the required radiographic exposure time, while remaining accurate to within a few per cent. Simple counters were unable to meet these requirements unless undesirably large apertures were permitted.

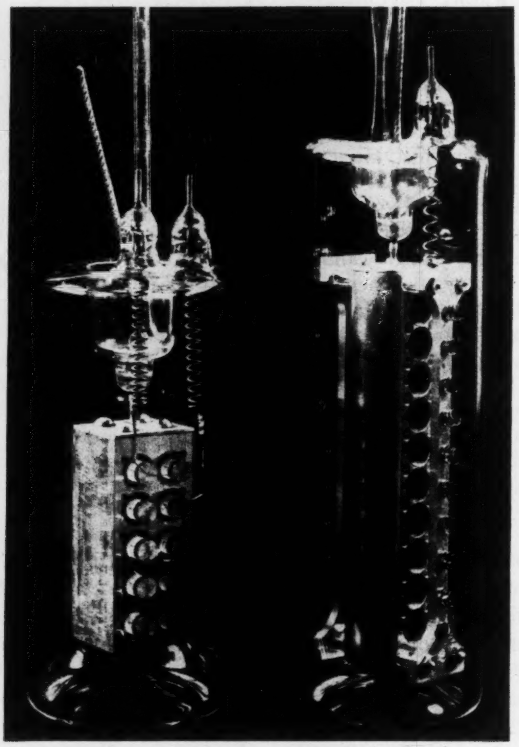
(a) (b)

Fig. 1.—Counters for X-ray Exposure Meter.

Only a small fraction of the intensity of high-voltage X-rays and gamma rays is absorbed in the gaseous volume of a counter. The counter discharges are triggered by electrons released from the metal cathode by Compton scattering of the primary X-rays or gamma rays. The efficiency of this process is of the order of one electron ejected from the cathode wall per hundred incident gamma ray quanta, in a counter with a smooth-surfaced cathode. In radiography with gamma radiation and lead intensifying screens, somewhere between 10^8 and 10^9 quanta per square centimeter are required to produce good film blackening. A simple counter may measure the above energy with an error of only 0.1 per cent in 1 sec. if its aperture is 1 sq. cm. However, for X-ray radiography in the region of 200 kv., the employment of chemical intensifying screens, such as calcium tungstate, decreases the duration of photographic exposures to such an extent that a simple counter of small aperture becomes relatively too insensitive for use in the exposure meter. To obtain the desired sensitivity while keeping the diameter of the measured beam of radiation small, the counter of Fig. 1 (*b*) was designed. This counter has worked satisfactorily with an aperture of only $\frac{1}{4}$ in. in diameter.

The counter is essentially a block of ten small counters connected in parallel. Brass-mesh cylinders, mounted in a duralumin block, form the cathodes. The anode wires are tungsten, 5 mils thick, and are held taut by springs. The guides that center the wires are supported by glass insulators. An alternative construction, Fig. 1 (*a*), employs punched mica disks for centering the wires, and glass capillary tubing for spacing the springs and leads of the wire system from the cathode block. Such tubes are more easily built than that shown in Fig. 1 (*b*).

The substitution of 100-mesh cathodes for the smooth-wall type has been found to double approximately the counter efficiency.[3] Since the efficiency of absorption of hard radiation in the counter cylinder is so low, it is obvious that ten cylinders in the path of the radiation will absorb almost ten times as much as one cylinder. The combined use of mesh and a multicylinder arrangement gives a counter that has the requisite speed of response and accuracy for the X-ray exposure meter.

When working with no-screen (direct-exposure) film, a multiple counter is much more sensitive than is necessary. The sensitivity may be reduced by further limiting the aperture and by providing a means for cutting out all but one or two of the cylinders as in Fig. 1 (*a*).

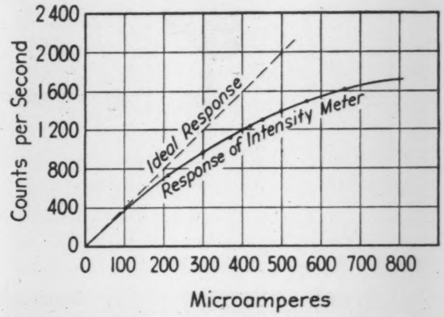

FIG. 2.—Response Curve of Multivibrator Circuit for Random Pulses.

A multivibrator circuit was chosen to amplify and equalize the counter pulses. The uniform pulses from the multivibrator are fed to the grid of a pentode, that is biased to cut-off. The plate current flow resulting from these pulses is smoothed by a condenser and indicated by a microammeter. Three type 6SJ7 tubes are used, two in the multivibrator and one for the output tube. Stabilized B voltage is secured by means of two VR-150 tubes. High voltage for the counter is obtained from a 3000-v. neon sign transformer used with a stabilizer of the "degenerate stabilizer controlled amplifier" type[4] utilizing type 811 and 802 transmitting tubes.

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[3] R. D. Evans and R. A. Mugele, "Efficiency of Gamma-Ray Counters," *Review of Scientific Instruments*, Vol. 7, p. 441 (1936).

[4] F. V. Hunt and R. W. Hickman, "On Electronic Voltage Stabilizers," *Review of Scientific Instruments*, Vol. 10, p. 6 (1939).

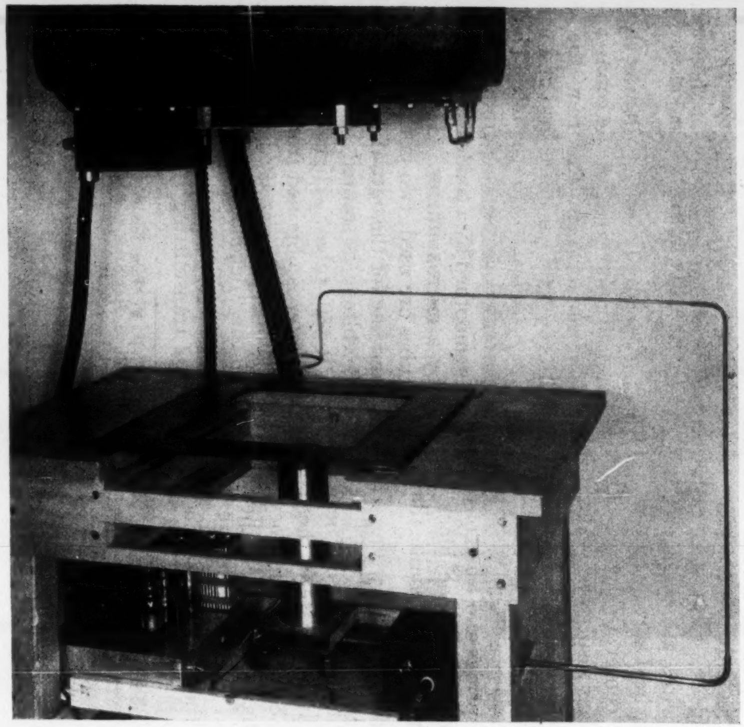

FIG. 3.—Arrangement of Exposure Meter Apparatus Used with 220-kv. X-ray Installation.

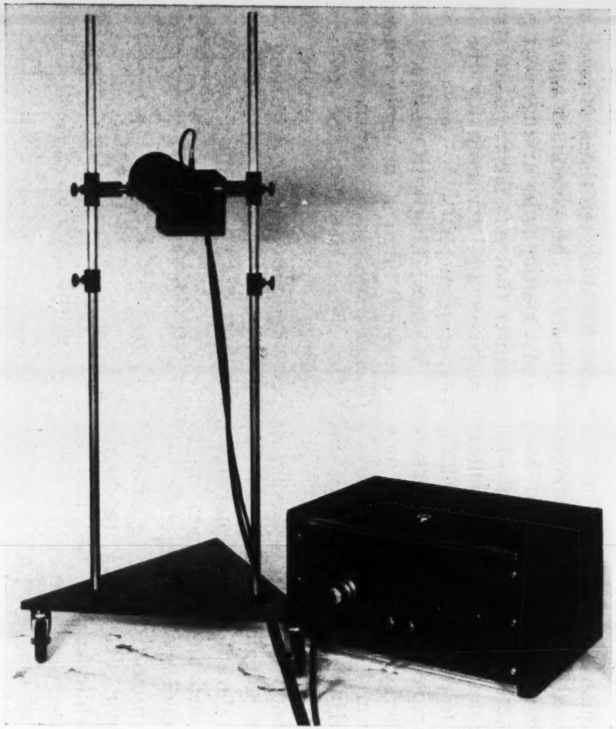

FIG. 4.—Exposure Meter for Use with Horizontal X-ray Beam.

The response of the intensity meter is shown in Fig. 2. The multivibrator circuit was designed for a resolving power of about 2000 pulses per second, sufficient to handle the intensities being measured.

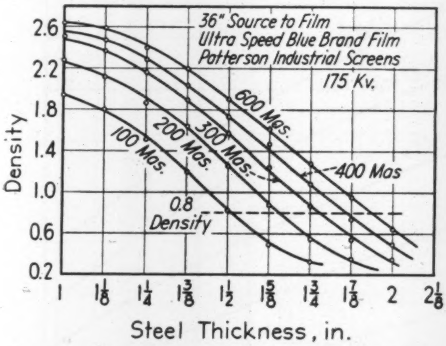

FIG. 5.—Dependence of Film Blackening on Thickness of Steel, for Various Exposures.

Figure 3 shows an arrangement in which the counter rests in a lead jug under the specimen table. The jug is covered by a lead cap, 1-in. thick with a ¼-in. hole drilled down its center, to define the aperture. Specimens to be radiographed are supported on the mask above the counter and the position of the counter aperture with respect to the specimen surface is indicated by an arm of brass tubing extending out from under the table and back over the top of the specimen. The jug, which is mounted on a dolly, can be moved about freely to permit exposure determinations through any portion of a casting of nonuniform thickness. The chassis seen under the table is similar to the circuit described above. The output meter is located on the control panel of the X-ray apparatus in an adjoining room.

For operation with a horizontal beam, the apparatus shown in Fig. 4 was built on a base equipped with casters, and designed to permit up and down, and tilting motions of the counter unit. The high-voltage and B supplies for the counter and multivibrator are contained in the cabinet at the right and the multivibrator unit itself is located in the box attached to the rear of the lead cylinder.

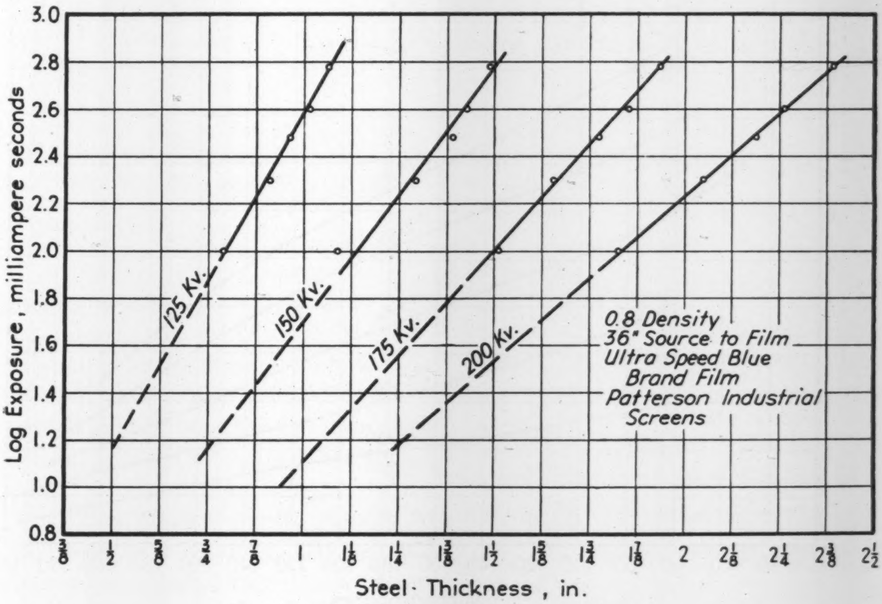

FIG. 6.—Form of Standard Technique Chart.

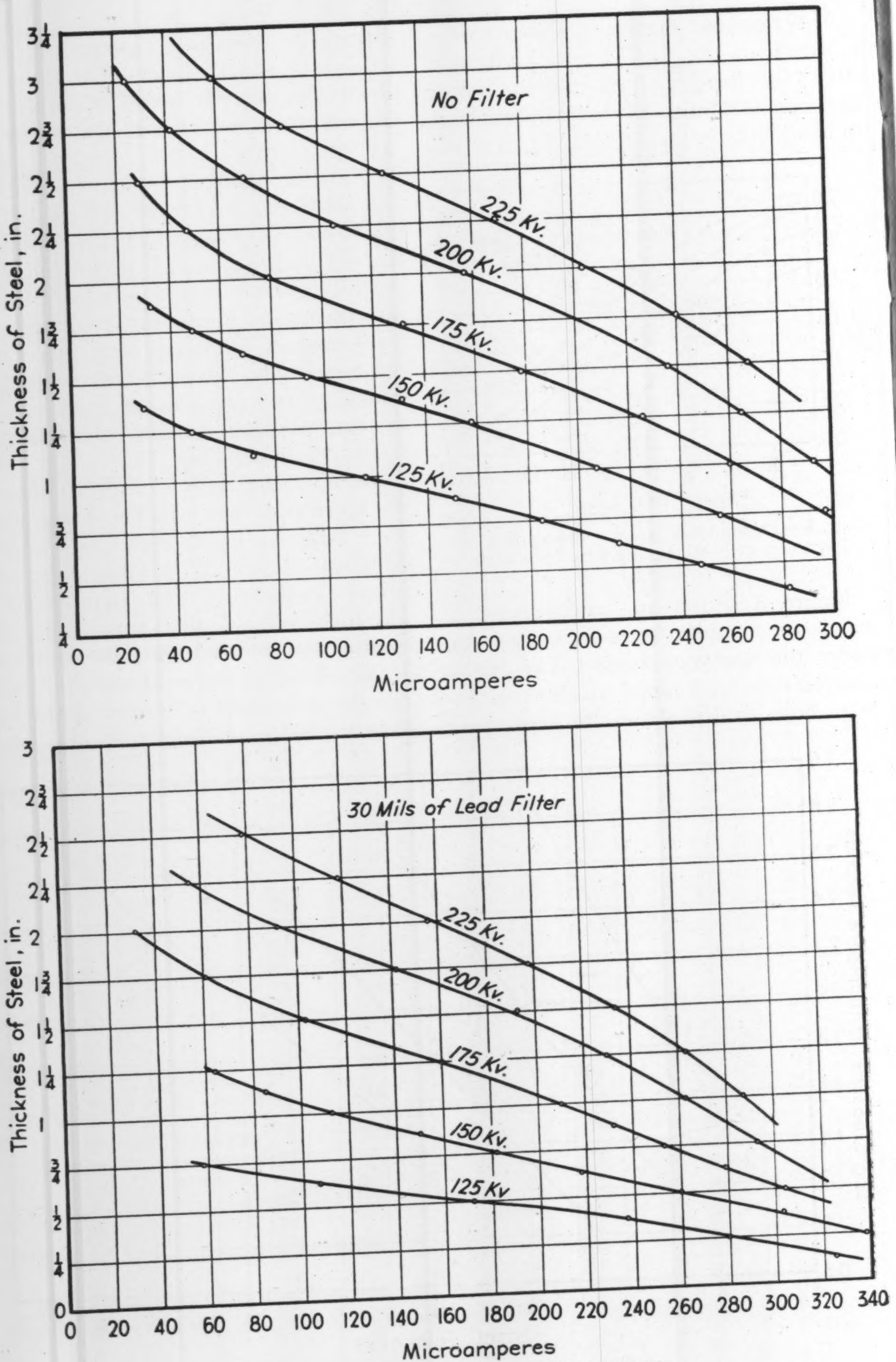

FIG. 7.—Relation of Meter Readings to Steel Thickness.

CALIBRATION

The calibration of the X-ray exposure meter was carried out in the following manner. A steel test block was employed, composed of steps of graded thickness, each step $\frac{1}{2}$ in. wide and $\frac{1}{8}$ in. higher than the preceding step. This block was radiographed with exposures of 100, 200, 300, 400, and 600 mas., at voltages of 125, 150, 175, and 200 kv. The radiographs exhibited a stepwise variation in blackening and the density of each step was measured with a densitometer. The results of the five exposures taken at a given kilovoltage are shown in Fig. 5. Families of S-shape curves, spaced logarithmically with respect to each other, were obtained. By plotting abscissae corresponding to a given density against the logarithm of the exposure, approximately straight-line curves, of the type shown in Fig. 6, are obtained. This is the form of the standard technique chart generally used. Next with the counter in its position below the film holder and with the X-ray tube at the same height as it was when the radiographs were taken, indications of the output meter were observed for the range of steel thicknesses generally covered at the five kilovoltages, Fig. 7.[5] Finally, the meter indications through various steel thicknesses were plotted, against the corresponding exposure times required to produce 0.8 density. The curves obtained at 125, 150, 175, and 200 kv. are drawn, in Fig. 8, in the form of a

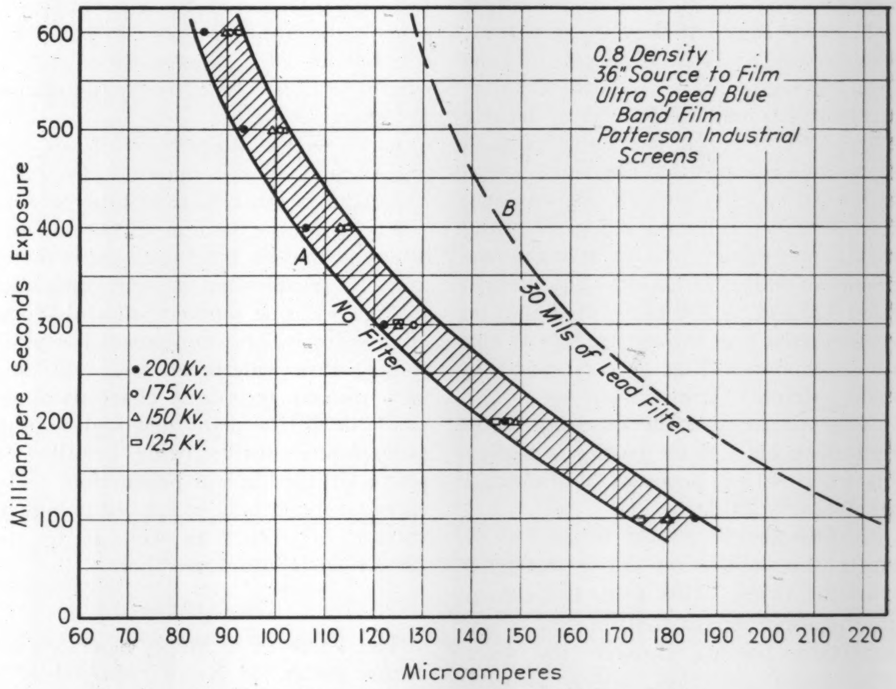

FIG. 8.—Calibration Curves for X-ray Exposure Meter.

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[5] The nonlogarithmic shapes of the curves may be understood from the calibration curve of Fig. 2. At zero steel thickness the curves reach the maximum counting rate of the multivibrator circuit.

band. This is the calibration curve of the exposure meter for the particular type of film used.

DISCUSSION OF DATA

From the material presented, it is immediately apparent that there are many advantages to be gained by use of the exposure meter in routine industrial and in research radiography. The advantages may be summarized as follows:

1. From Fig. 6 it may be seen that, for example, at $1\frac{7}{8}$ in. of steel, a change from 175 kv. to 200 kv. requires a change of about 400 mas. to maintain a density of 0.8. On the other hand, the exposure meter indications, Fig. 8 are, for practical purposes, independent of the voltage. For example, 127 microamperes on the meter corresponds to an average exposure of 300 ± 25 mas. for any voltage from 125 kv. to 200 kv. (It should be remembered that the four points at any given exposure, Fig. 8, correspond to widely different steel thicknesses.)

The variation of the exposure meter indication for a given density is negligible for practical purposes in the range from 125 to 200 kv.

2. The use of a lead filter, Fig. 8, shifts the calibration curve to higher counting rates. This is due to a change in the wave length distribution of the radiation, passing through the filter. The softer radiation, which is most efficient in blackening the film, is very effectively absorbed by the lead. For corresponding exposures, the no-filter curve is separated from the 30-mils lead filter curve by what amounts to about 0.4 in density units. Calibration curves for materials such as copper and brass fall between curves *A* and *B* of Fig. 8, and do not differ much from those for steel. For general radiography a single average calibration curve may be drawn that will assure a density between 0.7 and 1.2 for any material and for any kilovoltage.

3. Since the meter is read with the counter always at the film position, proper exposure is indicated for any geometry of the radiographic set-up.

4. The small aperture of the counter adapts it to scanning portions of a specimen where the thickness is not uniform. The meter indications may then be interpreted to tell whether a single exposure will prove satisfactory, whether use of a filter is called for, or whether more than one exposure is necessary.

5. At every appearance of an improved film on the market, new technique charts must be established for all voltages, source to film distances, and filter thicknesses, but the exposure meter need be recalibrated at only one kilovoltage and one source-to-film distance. High-voltage X-ray equipment is rapidly coming into widespread use, and only very meager technique chart data are available for the many new high-voltage radiographic outfits being installed in plants engaged in war production. The exposure meter may be applied to higher-voltage apparatus as well as to the 220-kv. installation used here.

CONCLUSION

The value of a simply operated exposure meter for X-ray radiography is apparent to any radiographer. In the rush of wartime work, radiographs of poor density are being accepted because of the expense of film and time. The efficiency of the X-ray exposure meter described in this paper has been proven in daily use at the Naval Research laboratory, both in routine inspection and in research. In a recent problem, it was required to radiograph fifty steel plates of different thicknesses and obtain uniform density in all the radiographs. By employing the exposure meter, a set of radiographs was obtained of which only two deviated by as much as 5 per cent from the desired 0.8 density.

The material of this paper has been confined to a description of the X-ray exposure meter. In gamma radiography the problem is much simpler, since the wave length distribution of radiation emitted by the source does not vary. The gamma-ray slide rule has proven satisfactory so long as the thickness of specimen material is accurately known. However, when castings of complicated structure are radiographed, it becomes difficult to correctly estimate the thickness of material in the path of the radiation. Figure 9 shows a gamma-ray exposure meter designed so that the sensitive element, a simple counter, may be held against the film holder while the correct exposure is read from the milliammeter. This may be done after the exposure has been started. The counter is sufficiently small so that all portions of the film holder may be scanned to determine the over-all best exposure for specimens of varying thickness.

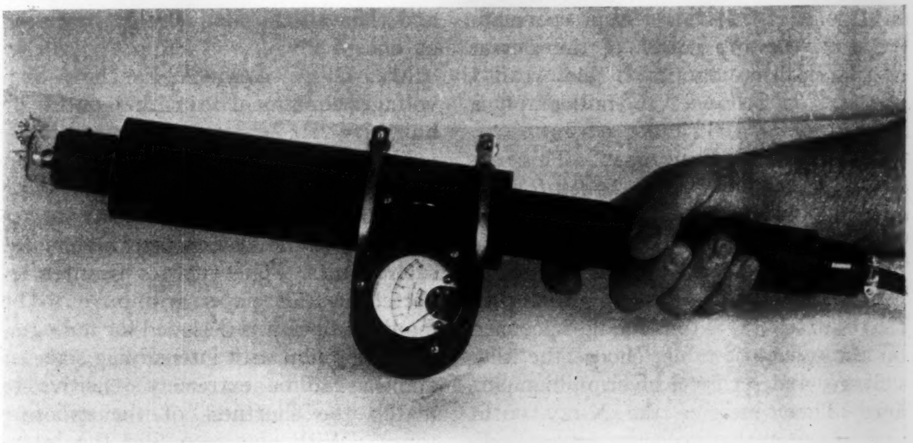

FIG. 9.—Exposure Meter for Gamma-ray Radiography.

The X-ray exposure meter circuit is designed for 110-v. a-c. operation. The gamma-ray meter circuit can be built for battery operation to facilitate its use in the field.

DISCUSSION

Mr. H. H. Lester.[1]—Will you elaborate on the application of this device in practical radiography? How can I use it, for instance, to radiograph a steel plate?

Mr. Herbert Friedman.[2]—This particular meter has been designed for use with 220-kv. equipment, and we have adjusted its sensitivity to cover the range of steel thicknesses generally radiographed with such apparatus. All that is necessary is to place the specimen on the specimen table, choose the kilovoltage, and set for a given milliamperage. Then energize the X-ray outfit and observe the exposure meter indicator. From the calibration curve, you immediately obtain the exposure necessary for the desired density.

Mr. Lester.—Would it not be simpler to take a couple of trial exposures?

Mr. Friedman.—That would require that you process the film after each exposure. Let us assume this would take about 10 to 15 min. If you then find your estimated exposure was not sufficiently close, further trials are necessary. With the exposure meter, you have an exact determination and consequently a successful exposure on every attempt.

If the radiographer is at all uncertain of his estimated exposures, he must process each film directly following its exposure. With the meter, a large number of specimens may be radiographed; and then all the film may be processed at once.

Mr. C. A. Adams.[3]—For how high voltage equipment can this outfit be built?

Mr. Friedman.—We have not yet had an opportunity to try it on anything like million-volt equipment, but we are planning to build exposure meters for such outfits. The type of counter reported in this paper will have to be modified since it is designed for radiography using film with intensifying screens, and has to be extremely sensitive to match the shortness of the exposure times. With no-screen film the intensity required to blacken the film is so much greater that a very simple type of counter can be used and still give the required speed of response on the meter.

Mr. L. W. Ball.[4]—I should like to make two suggestions. I think several of us will question the need for the apparatus for the purpose you have suggested. We can predict the exposure sufficiently accurately for ordinary work, although if we were using a density of 0.8 we would have to be rather more concerned.

These are the two suggestions. First, when you are working with radon you are much more likely to have an unknown exposure, and I think your meter would be of value to anyone who is working with radon.

Secondly, if you were working with

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[1] Principal Physicist, Watertown Arsenal, Watertown, Mass.

[2] Associate Physicist, Division of Physical Metallurgy, Naval Research Laboratory, Anacostia Station, Washington, D. C.

[3] Professor of Engineering, Emeritus, Harvard University, Cambridge, Mass.; and Consulting Engineer, Edward G. Budd Manufacturing Co., Philadelphia, Pa.

[4] Research Physicist, Triplett & Barton, Inc., Burbank, Calif.

photographic papers instead of film the latitude is very, very much less, and the usefulness of your instrument would be very much greater.

MR. FRIEDMAN.—We have been designing a meter for radon, or possibly radio-yttrium, which has a still shorter half life. If an exposure meter is available for use with these sources it is not necessary to know how far the source has decayed. A meter for these hard radiations is fairly simple to construct.

MR. OTTO ZMESKAL.[5]—Mr. Friedman has no doubt made measurements of the sensitivity of defect detection?

MR. FRIEDMAN.—In one of the curves shown, you may have seen that a thickness change of the order of 1 per cent at 125 kv. produces almost a 100 per cent change in the meter indication. It would be difficult to miss a 1 per cent change in effective thickness in scanning, with a $\frac{1}{4}$-in. aperture. The sensitivity of the counter in measuring effective thickness is better than that of any other known device. It far exceeds that of a photographic film. We have been working on the problem of developing Geiger-Muller counter devices for use with radium or with X-rays in inspection. The counter is very effective where you have a problem of measuring effective wall thickness or some general, extensive porosity. It fails where you have a fine crack or a cavity whose diameter measures much less than the aperture of the counter.

MR. ALEXANDER GOBUS[6] (*by letter*).—The development of a device of such sensitivity as to act as an exposure meter for radium radiography is of utmost importance. Mr. Friedman indicated in verbal discussion that he had such a project under way and that preliminary tests were satisfactory.

One very important use would be in the radiography of piping and large cylinders.

It should enable one to scan the entire circumference of the object. This would show if the capsule were centered. If the operator had other positive means of centering, then differences in wall thickness would be indicated and the radium so moved as to give a uniform reading around the entire object.

Thus, through the elimination of density differences, now not a positive accomplishment, radiographers everywhere should benefit.

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[5] Assistant Professor of Metallurgy, Illinois Institute of Technology, Chicago, Ill.

[6] Chief Metallurgist, Lucius Pitkin, Inc., New York, N. Y.

A STUDY OF CASSETTE DESIGN FOR THE RADIOGRAPHY OF AIRCRAFT CASTINGS

BY L. W. BALL[1]

The apparatus commonly used for radiographing aircraft castings and forgings was inherited from medical diagnostic radiography. One part of this inheritance was a cassette design consisting of bakelite, felt, fluorescent screens, and a brass frame. Such a design is most unsuitable for aircraft radiography. This fact probably has been realized by the many industrial radiographers who have been using aluminum front cassettes, but present restrictions on supplies may re-introduce objectionable bakelite fronts or other detrimental arrangements in homemade cassettes.

The features that are essential to a well-designed cassette are partly mechanical and partly concerned with scattered X-radiation.

The purpose of this paper is to provide a systematic demonstration of the features that are essential to prevent scattered radiation from the cassette material from spoiling radiographs.

SCREEN FILM CASSETTE DESIGN

Because it is necessary to provide for very close contact between the screens and the film, a frame with a rigid periphery is essential. This periphery should be constructed from material that is relatively opaque to X-rays. If bakelite or any other radiographical transparent material is used, sideways transmitted scatter will fog the edge of the film. Figure 1(*a*) shows an experimental arrangement devised to demonstrate this factor. One exposure was made with a bakelite bar representing a section of the cassette periphery and another with a brass bar. The results could be compared by visual examination of the sensitivity at the edge of the image of the slotted penetrameter block. However, reproduction of radiographs alters the apparent sensitivity so greatly that photometer curves provide a preferable means of illustration. Figure 1(*b*) shows photometer traces made across the 15, 10, and 5 per cent slots at $\frac{1}{4}$ and 1 in. from the edge of the cassette. The fog from the bakelite reduces the sensitivity at $\frac{1}{4}$ in. five times, and at 1 in. twice. No fog was observed from the brass.

The exposures used in this and several subsequent experiments were the normal exposures for penetrating 1 in. of duralumin. At the same kilovoltage, 100 times the exposure would be required to penetrate each extra inch of duralumin; that is, at the same kilovoltage the loss in sensitivity would be one hundred times greater for 2 in. of duralumin, 10,000 times greater for 3 in., and so on.

The materials forming the front part of the cassette and producing forward transmitted scatter, and the materials forming the back part of the cassette and producing back scatter, may be considered separately. In each case, scattering of the intense primary beam by the cassette material will be concentrated at the edges of any image, and its effect will be to give a lower sensitivity at $\frac{1}{4}$ in. than at 1 in. from the edge.

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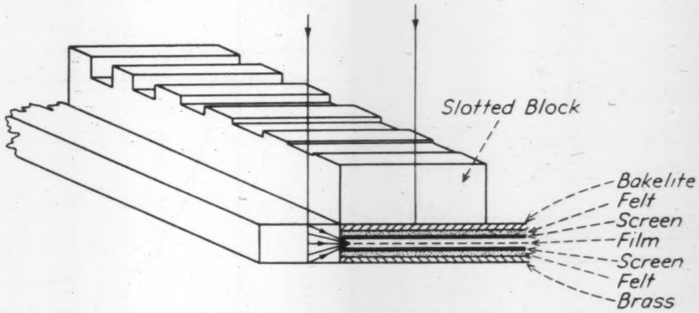
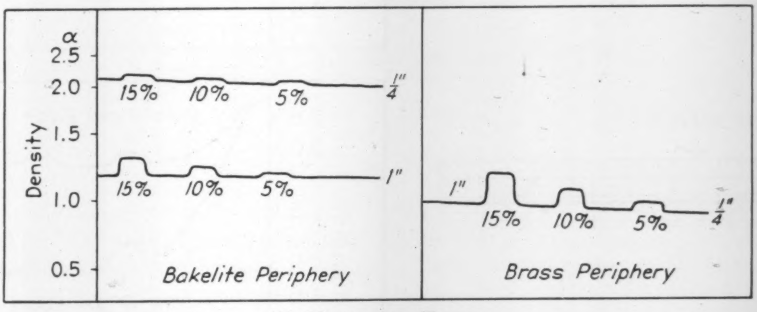
[1] Research Physicist, Triplett & Barton, Inc., Burbank, Calif. Formerly with the National Research Council of Canada.

By combining a back lead screen and immediate contact between a penetrameter and the film, we have what may be called a "no cassette scatter" arrangement. With this arrangement the sensitivity at $\frac{1}{4}$ in. from the edge is equal to the sensitivity at 1 in. from the edge.



(*a*) Experimental Arrangement.



(*b*) Photometer Traces.

FIG. 1.—Scatter from Cassette Periphery.

The experimental arrangements shown in Fig. 2 gave the results shown in the corresponding photometer curves. Figure 2(*a*) shows that the screen material produces negligible scatter with an exposure penetrating 1 in. of duralumin. Figure 2(*b*) shows that the $\frac{1}{8}$ in. bakelite causes a three times loss in sensitivity at $\frac{1}{4}$ in., and Fig. 2(*c*) shows that raising the bakelite from the film by the introduction of $\frac{1}{8}$ in. of felt increases this loss to six times.

The arrangement of Fig. 2(*c*) is one likely to be used in homemade cassettes. It should be rigorously suppressed for use in the radiography of aircraft castings or forgings.

Two methods of reducing the forward transmitted scatter from the cassette front should be incorporated in a good cassette design. First, the scattering material should be brought as near to the film as possible (compare Fig. 2(*b*) with (*c*)). This requires a thin front screen plus complete elimination of the felt or other material between the front and the screen.

Secondly, thin sheet metal should be substituted for the bakelite. Previous

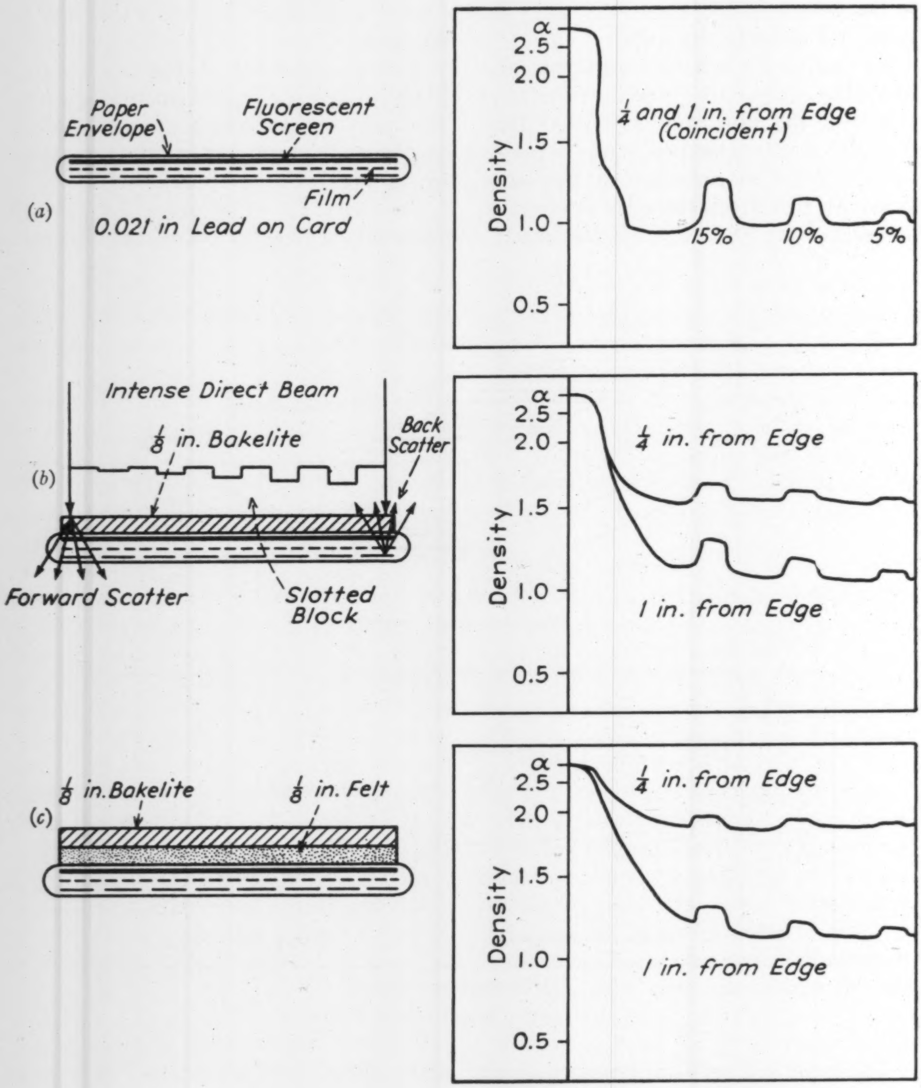


FIG. 2.—Screen Film—Forward Scatter.

experiments have shown that aluminum produces roughly the same amount of forward transmitted scatter as the same weight of bakelite.[2] But an aluminum alloy cassette front using the stretched-skin type of construction need be only one-third as heavy as bakelite. The advantage of the reduction in the weight of the scattering material and the closer approach to the film make thin aluminum fronts much superior to bakelite.

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[2] L. W. Ball, "A Study of Secondary Radiation in Relation to the Radiography of Aircraft Castings," ASTM BULLETIN, No 115, March, 1942, p. 27.

Steel 0.005 to 0.010 in. thick is satisfactory if suitable methods of fabrication are available.

When these features are embodied in the cassette design, forward scattering from the cassette material is not appreciable with the normal exposure for penetrating 1 in. of duralumin. To provide testing conditions equivalent to the most severe ones occurring in normal aircraft radiography, a slotted block penetrameter 3 in. thick was used.

Before measuring the effect of X-ray scatter with this penetrameter, the experimental arrangement shown in Fig. 3 was devised to measure the effect of light halation. In one exposure actinic light produced by that part of the fluorescent screen exposed to the intense direct X-rays might have spread into the image of the penetrameter. In another exposure, made with the black paper threaded right through the film and the screen, light halation could not occur.

Photometer curves obtained in this experiment proved that at ¼ in. from the edge of the intense light a slight decrease in sensitivity due to light halation could occur. This deterioration was negligible when the contact between the fluorescent screen and the film was perfect, but with a relatively low pressure a decrease of 30 per cent was recorded.

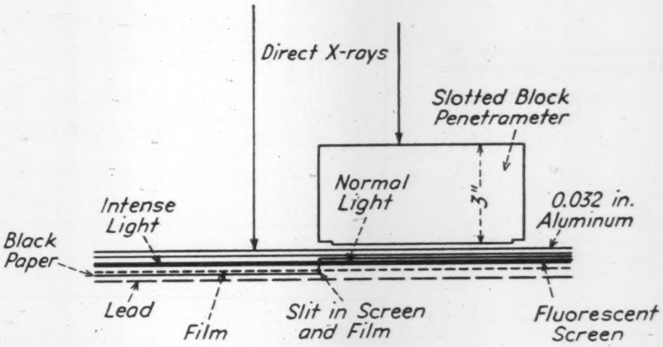


FIG. 3.—Light Halation.

Figure 4(*a*) illustrates a cassette designed for minimum forward scatter. The corresponding photometer curves show that the sensitivity at ¼ in. from the edge is still three times less than at 1 in. for the normal exposure used to penetrate 3 in. of duralumin.

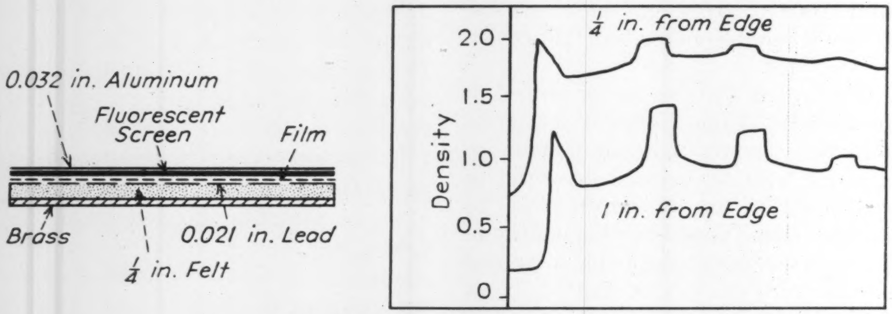
Back scatter from the cassette material behind the film produces results very similar to those for forward scatter if the same materials are used. But in the radiography of light alloy castings, it is neither desirable nor practicable to use a cassette back that transmits X-rays. A good cassette design places opaque material as near as possible to the back of the film.

Three experimental arrangements for testing back scatter together with the results are shown in Fig. 4. Comparison of these photometer curves demonstrates the advantage of closing the gap between the film and the opaque material behind it.

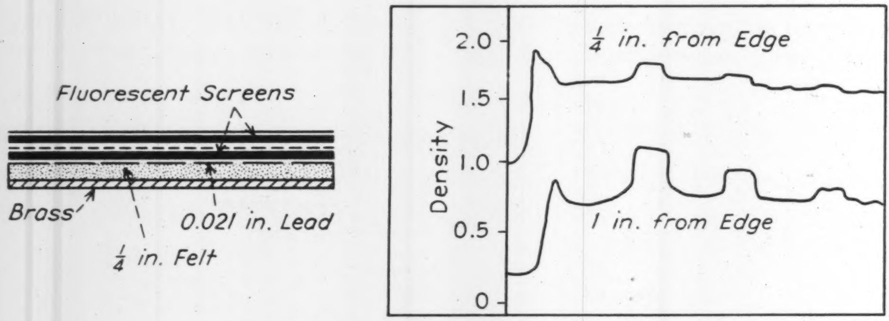
DIRECT-EXPOSURE FILM CASSETTE DESIGN

A series of experimental tests using direct-exposure film were made with the arrangements, and the results shown in Fig. 5.

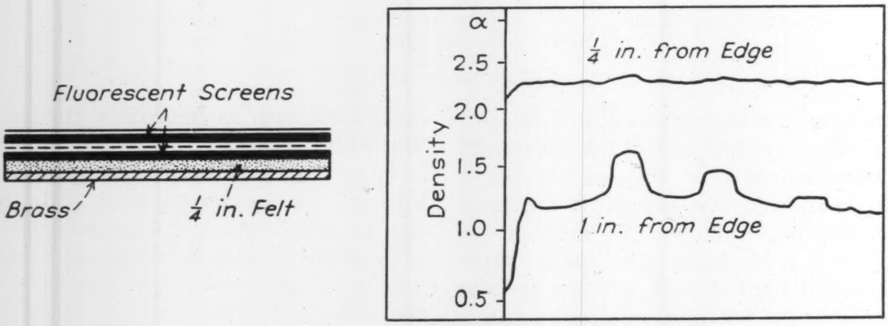
Comparison between Fig. 5 (*a*) and (*b*)



(*a*) Opaque material in contact.



(*b*) Opaque material close to film.

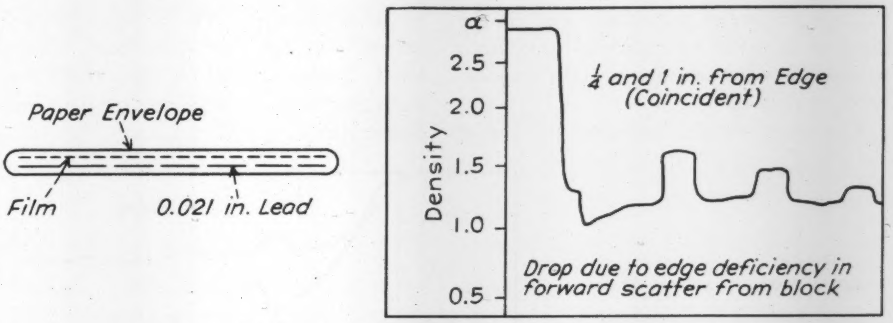


(*c*) Opaque material away from film.

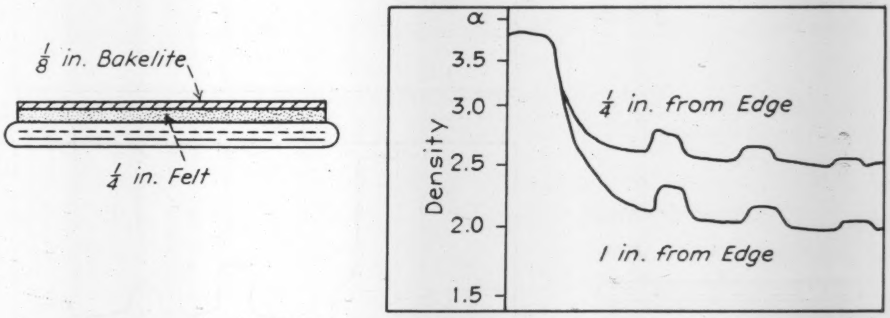
FIG. 4.—Screen Film—Back Scatter.

shows that bad forward transmitted scatter conditions reduced the sensitivity two times at ¼ in. from the unblocked edge. This decrease in sensitivity is less than for the same conditions with screen film because the emulsion contrast of this film increases considerably with increasing density.

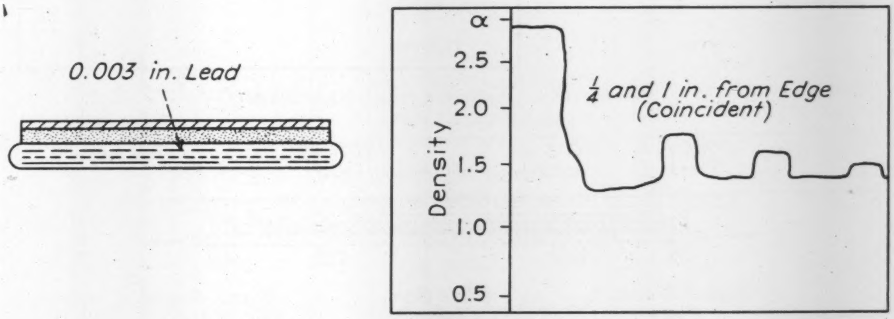
The effect of the 0.003 in. thick front lead Fig. 5(*c*) was to restore the sensi-



(*a*) No cassette scatter.



(*b*) Bad forward scatter.



(*c*) Forward scatter with front lead screen.

FIG. 5.—Direct Exposure Film—Forward Scatter.

tivity to 0.9 times that of the "no cassette scatter" arrangement.

With direct film, cassette back scatter should be eliminated completely by a thick back lead screen. About 0.018 in. of lead provides a suitable flexibility for mounting on cardboard in the manner described by Tobey and Seemann.[3] An extra 0.003 in. of antimony lead in the form of a cover provides a convenient hard surface for cleaning and for replacement, if damaged.

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[3] R. G. Tobey and H. E. Seemann, "Mounting Lead Foil for Radiographic Screens," *Metals and Alloys*, July, 1940.

The experiment illustrated by Fig. 6 was made to demonstrate that a 0.021-in. back lead screen prevents even the worst back-scattering material[3] from spoiling the film.

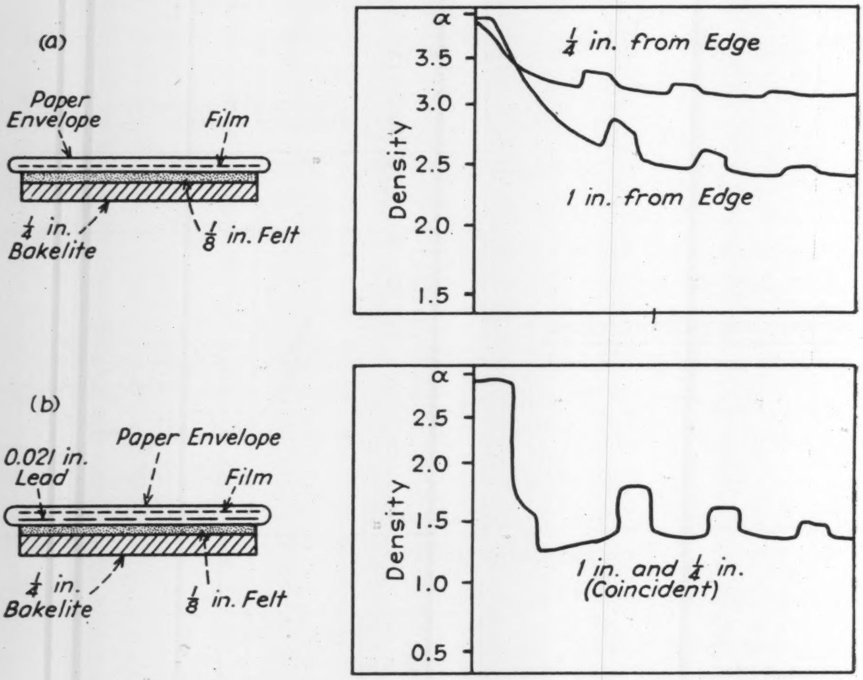

FIG. 6.—Direct Exposure Film—Back Scatter.

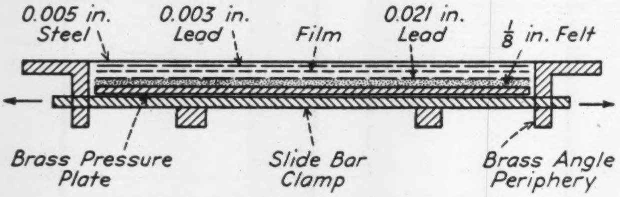
0.005 in. Steel
0.003 in. Lead
Film
0.021 in. Lead
$\frac{1}{8}$ in. Felt
Brass Pressure Plate
Slide Bar Clamp
Brass Angle Periphery

FIG. 7.—National Research Laboratory Cassette Arranged for Direct Exposure Film.

The arrangement illustrated by Fig. 7 includes all the features by which scatter from the cassette material can be reduced to a minimum. To provide a very severe test for this cassette, a slotted block penetrameter 4 in. thick was radiographed; cassette scatter was quite negligible.

RELATION OF CASSETTE DESIGN TO RADIOGRAPHIC PRACTICE

A radiographer has to consider cassette design in relation to two important duties: First in the selection and purchase of his equipment, and second in the selection of calibration techniques that are suitable for each class of work in which he is engaged.

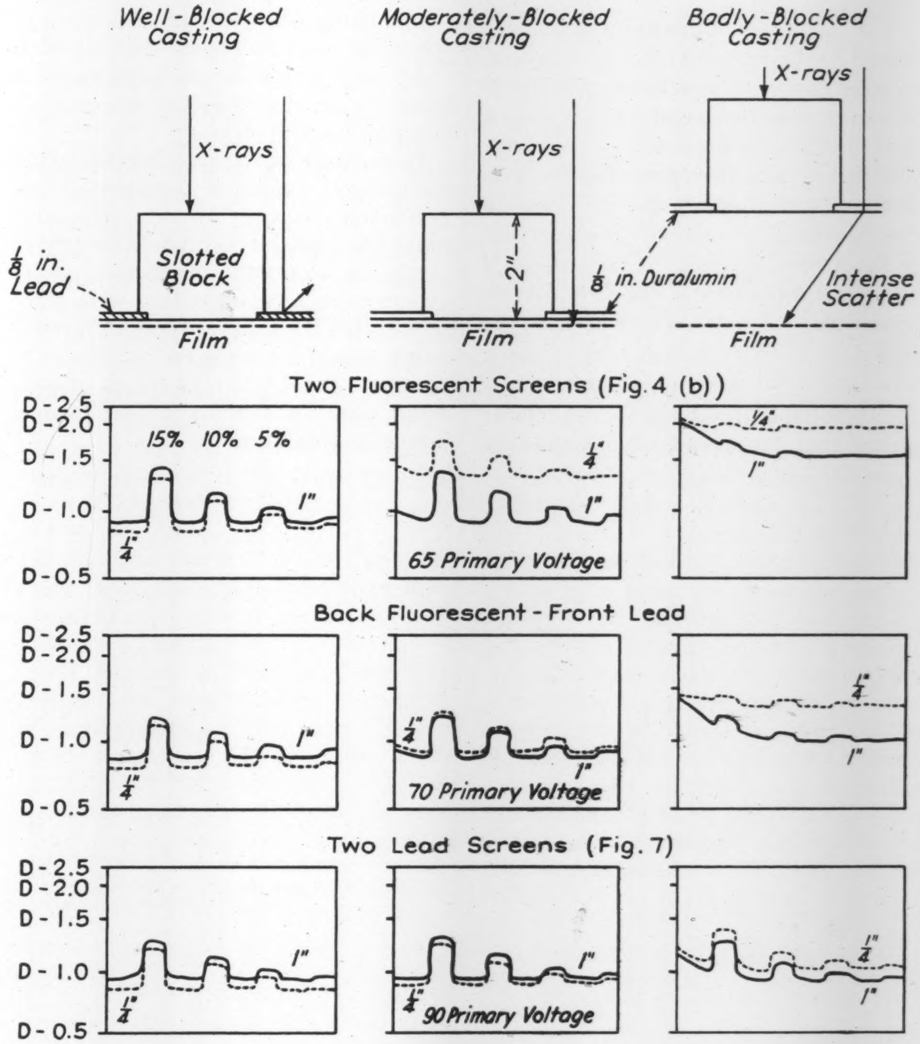

FIG. 8.—Relation of Cassette Design to Casting Scatter Group.

Different cassettes, together with their screens, can be compared for speed, sensitivity, and range by preparing a few radiographs of a slotted wedge penetrameter.[4] Tests of resolving power can be made with a narrow slit penetrameter, and a check on screen contact can be made with the wire-mesh test. Comparative tests of the degree to which scattered X-rays will spoil the sensitivity at the edge of the radiographic image should be made by the slotted block and photometry method described in this paper.

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[4] L. W. Ball, "Technique and Organization in the Radiography of Aircraft Castings," Topical Discussion on Radiographic Testing of Airplane Components, issued in mimeographed form by Am. Soc. Testing Mats. (1940).

The choice of calibration exposures should be based primarily on a general consideration of scattered radiation.[2] However, cassette design is an important part of this consideration.

Figure 8 has been prepared to demonstrate the importance of using a cassette design suitable to the object that is being radiographed. Comparison of the three exposures in the first column shows that for well-blocked objects, the highest sensitivity is obtained by using two fluorescent screens. Comparison of the three exposures in the second column shows that for moderately blocked objects, the speed of the back fluorescent screen can be used to advantage, but a front fluorescent screen reduces the sensitivity at the edges.

Comparison of the three exposures in the third column shows that a thick front lead screen is essential to even moderate reliability in the examination of badly blocked objects.

In the past two years, progress in the casting and forging of complicated and previously built-up aircraft structural parts has placed many vital patterns in the badly blocked radiographic group. In some cases, cassettes that were satisfactory for the simpler types of aircraft parts should be replaced or rebuilt to eliminate the deleterious effects of scattered radiation that have been demonstrated in this paper.

DISCUSSION

Mr. H. H. Lester.[1]—I should like to ask Mr. Ball how he would block a cylindrical casting, or something in the shape of a round cold-rolled bar.

Mr. L. W. Ball.[2]—On the cylindrical bars we can expect trouble from cassette scatter at the edge because the intense direct radiation is passing by there, and we can expect a little trouble due to scatter from the periphery of the bar going into the center. If it is a simple light-alloy bar, we immerse it in carbon tetrachloride. If it was part of a complex pattern, then we would switch over to two lead screens and a higher voltage. We would sooner have 2 per cent sensitivity throughout the object than have ½ per cent sensitivity in one part and only 5 per cent in another.

Mr. Herman E. Seemann.[3]—In connection with cassette design, I presume it is well understood that the rigidity of the front and back plates is an important factor in making satisfactory contact between screens and film. In connection with Mr. Ball's paper I should like to raise the question as to whether he can, from the existing data, select a material which will keep the scatter to a minimum and still maintain the necessary rigidity. Different materials have different elastic constants, so different thicknesses would be required for the same rigidity.

Mr. Ball.—If you are taking a plain sheet and screwing it down on a frame, then bakelite does have the tremendously attractive virtue of rigidity. If you take some soft aluminum foil, and screw it down there will not be sufficient rigidity to give good screen contact or to support the radiographic subject. If you can get a hard alloy and stretch it over the edges and rivet it like an airplane wing, you can effectively increase the modulus of elasticity. We tried to conserve metals by using zinc sheets from a multigraph machine. They had a suitable filtration and low X-ray scatter, but the zinc was so soft that after a few days the cassette front was like an ocean wave.

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[1] Principal Physicist, Watertown Arsenal, Watertown, Mass.

[2] Research Physicist, Triplett & Barton, Inc., Burbank, Calif. Formerly with the National Research Council of Canada.

[3] Physicist, Eastman Kodak Research Laboratories, Eastman Kodak Co., Rochester, N. Y.

EQUIVALENT PENETRAMETERS IN RADIOGRAPHIC TESTING

By Robert J. Schier[1] and Gilbert E. Doan[1]

Synopsis

The possibility of using penetrameters next to the film, instead of next to the source, in radiographing pipes is explored and the thickness of these penetrameters to make them equivalent to those used in the A.S.M.E. code is determined experimentally.

Welded pipe joints are difficult to radiograph when the diameter is small because the penetrameter required by the A.S.M.E. Code[2] must be placed on the *inside* of the pipe. Cases have been known where a hole was cut into the pipe near the joint so that the penetrameter could be inserted. The hole was later welded shut but not radiographed, making the whole procedure an apparent absurdity.

In difficult cases of this kind a substitute penetrameter might be used for the standard, the substitute being placed on the *outside* of the pipe next to the film. The dimensions of this substitute penetrameter would have to be such that it was equivalent, when placed on the outside of the pipe, to the standard penetrameter placed inside the pipe. The development of an "equivalent penetrameter" is the purpose of this study. Wall thicknesses of $1\frac{1}{2}$, $1\frac{3}{4}$, and 2 in. were used. Gamma rays only were available. Since they will not give the 2 per cent sensitivity required by the A.S.M.E. Code at less than $1\frac{1}{2}$-in. thicknesses, thinner sections than $1\frac{1}{2}$ in. were not used.

Experimental Work

The source of gamma radiation was a spherical duralumin capsule containing 25 mg. of radium sulfate. The internal and external diameters of the capsule were 3.0 and 4.0 mm., respectively.

The test specimens were made of mild steel blocks $1\frac{1}{2}$, $1\frac{3}{4}$, and 2 in. thick. Penetrameters as specified in the A.S.M.E. Code[2] were located on the surfaces of the specimens nearest the source. On the backs of the specimens adjacent to the films were located thinner penetrameters containing the same size holes as the standard penetrameters but with thicknesses decreasing down to 0.003 in. Plate thicknesses, source-to-film distances, and penetrameter sizes are listed in Tables I to VI. The source-to-film distances used correspond to vertex angles of approximately $1\frac{1}{2}$ and 1 deg.[3]

The specimens were mounted on wooden stands and grouped around the source in the manner shown in Fig. 1. The front surfaces of the specimens were

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[1] Gotshall Scholar in Metallurgy, and Professor of Metallurgy, respectively, Lehigh University, Bethlehem, Pa.

[2] "Unfired Pressure Vessels," A.S.M.E. Code, p. 58 (1940).

[3] Robert J. Schier and Gilbert E. Doan, "Precision Radiography—III," see p 1171.

TABLE I.—OBSERVED VISIBILITY OF EQUIVALENT PENETRAMETERS AS COMPARED TO A STANDARD PENETRAMETER.

Specimen thickness, 1½ in. Film 3B. Source-to-film distance, 9 in.

Penetrameter Thickness, in.	Penetrameter Location	Visibility Index of Penetrameter Holes		
		0.060 in.[c]	0.090 in.	0.120 in.
0.030	S[a]	4[b]	4	4
0.025	F[a]	6	6	6
0.020	F	5	5	5
0.0142	F	3	4	4
0.010	F	2	3	3
0.0075	F	0	1	2
0.005	F	0	0	1
0.004	F	0	0	0
0.003	F	0	0	0

[a] Source and film surfaces, respectively.

[b] Numbers indicate relative visibility, 1 and 6 being poor and good, respectively.

[c] Nearest drill sizes were used for holes.

TABLE II.—OBSERVED VISIBILITY OF EQUIVALENT PENETRAMETERS AS COMPARED TO A STANDARD PENETRAMETER.

Specimen thickness, 1½ in. Film 3A. Source-to-film distance, 11¾ in.

Penetrameter Thickness, in.	Penetrameter Location	Visibility Index of Penetrameter Holes		
		0.060 in.	0.090 in.	0.120 in.
0.030	S[a]	4	4	4
0.025	F[a]	5	5	5
0.020	F	4	4	4
0.0142	F	3	3	4
0.010	F	0	1	2
0.0075	F	0	0	1
0.005	F	0	0	0
0.004	F	0	0	0
0.003	F	0	0	0

[a] Source and film surfaces, respectively.

TABLE III.—OBSERVED VISIBILITY OF EQUIVALENT PENETRAMETERS AS COMPARED TO A STANDARD PENETRAMETER.

Specimen thickness, 1¾ in. Film 4B. Source-to-film distance, 9⅝ in.

Penetrameter Thickness, in.	Penetrameter Location	Visibility Index of Penetrameter Holes		
		0.070 in.	0.105 in.	0.140 in.
0.035	S[a]	3	3	3
0.030	F[a]	6	6	6
0.025	F	5	5	5
0.020	F	4	4	4
0.0142	F	2	3	3
0.010	F	1	2	2
0.0075	F	0	1	1
0.005	F	0	0	0
0.004	F	0	0	0
0.003	F	0	0	0

[a] Source and film surfaces, respectively.

TABLE IV.—OBSERVED VISIBILITY OF EQUIVALENT PENETRAMETERS AS COMPARED TO A STANDARD PENETRAMETER.

Specimen thickness, 1¾ in. Film 4A. Source-to-film distance, 11½ in.

Penetrameter Thickness, in.	Penetrameter Location	Visibility Index of Penetrameter Holes		
		0.070 in.	0.105 in.	0.140 in.
0.035	S[a]	4	4	4
0.030	F[a]	6	6	6
0.025	F	5	5	5
0.020	F	4	4	4
0.0142	F	3	4	4
0.010	F	2	2	3
0.0075	F	0	2	2
0.005	F	0	0	0
0.004	F	0	0	0
0.003	F	0	0	0

[a] Source and film surfaces, respectively.

TABLE V.—OBSERVED VISIBILITY OF EQUIVALENT PENETRAMETERS AS COMPARED TO A STANDARD PENETRAMETER.

Specimen thickness, 2 in. Film 5B. Source-to-film distance, 9⅝ in.

Penetrameter Thickness, in.	Penetrameter Location	Visibility Index of Penetrameter Holes		
		0.080 in.	0.120 in.	0.160 in.
0.040	S[a]	5	5	5
0.035	F[a]	6	6	6
0.030	F	6	6	6
0.025	F	5	5	5
0.020	F	3	3	4
0.0142	F	0	1	2
0.010	F	0	0	2
0.0075	F	0	0	2
0.005	F	0	0	1
0.004	F	0	0	1
0.003	F	0	0	0

[a] Source and film surfaces, respectively.

TABLE VI.—OBSERVED VISIBILITY OF EQUIVALENT PENETRAMETERS AS COMPARED TO A STANDARD PENETRAMETER.

Specimen thickness, 2 in. Film 5A. Source-to-film distance, 11½ in.

Penetrameter Thickness, in.	Penetrameter Location	Visibility Index of Penetrameter Holes		
		0.080 in.	0.120 in.	0.160 in.
0.040	S[a]	5	5	5
0.035	F[a]	6	6	6
0.030	F	6	6	6
0.025	F	3	5	5
0.020	F	0	3	3
0.0142	F	0	1	1
0.010	F	0	0	1
0.0075	F	0	0	1
0.005	F	0	0	0
0.004	F	0	0	0
0.003	F	0	0	0

[a] Source and film surfaces, respectively.

oriented perpendicular to the direction of propagation of the rays.

The films were mounted in light-tight packs, each pack containing two films and three lead screens. A $\frac{1}{8}$-in. lead sheet served to give rigidity to the pack and to eliminate back scatter. The packs were securely fastened to the backs of the specimens with rubber bands. Eastman's Ultra Speed Duplitized Film was used, and exposure times were calculated from charts given by Barrett, Gezelius, and Mehl.[4]

Duplicate films were viewed superimposed, and a visual examination of the image densities was made to determine the equivalent thicknesses of the substitute penetrameters.

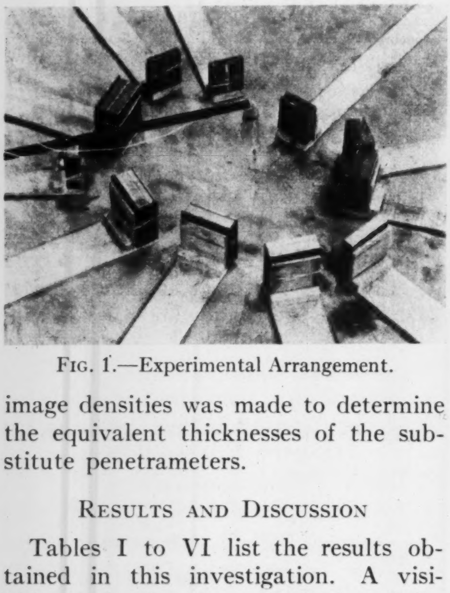

FIG. 1.—Experimental Arrangement.

RESULTS AND DISCUSSION

Tables I to VI list the results obtained in this investigation. A visibility index has been devised and included to facilitate a comparison of the results. The index indicates the relative ease with which the penetrameter holes were observed.

When the films were viewed, it was found that there was a slight difference between the sizes of some of the images of the equivalent penetrameters and those of the control penetrameters, but this is to be expected since registry of the equivalent penetrameters was almost entirely by the true shadow whereas the standard penetrameter gives umbral plus penumbral shadows.

Since the penetrameters were spread over the entire back surface of a specimen, not all of the penetrameters were in the same favorable location. The short focal distances used accentuated this effect. Hence the thicknesses given below are only approximate. They indicate that the thickness of an equivalent penetrameter increases as the specimen thickness increases, which is to be expected if the penetrameters are to indicate the same sensitivity in every case. The data showed that in order to be equivalent to the standard penetrameters for specimen thicknesses of $1\frac{1}{2}$, $1\frac{3}{4}$, and 2 in., the equivalent penetrameters should have, respectively, approximate thicknesses as follows:

0.0142 instead of 0.030, 0.020 instead of 0.030 and 0.025 instead of 0.040 in.

When an equivalent penetrameter is used, the radiographic conditions must of course be the same as those used when the penetrameter is located in its usual position on the specimen surface nearest the source.

Acknowledgments:

The authors gratefully acknowledge the generous assistance and cooperation of the Radium Chemical Co. and the Bethlehem Steel Co. The authors are also indebted for the fellowship under which this investigation was carried out provided by the endowment of Mr. William G. Gotshall.

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[4] C. S. Barrett, R. A. Gezelius, and R. F. Mehl, "The Technique of Radiography by Gamma Rays," *Metals and Alloys*, Vol. 1, p. 872 (1930).

DISCUSSION

Mr. R. W. Emerson.[1]—It may have sounded ridiculous when Mr. Doan mentioned that it was necessary to cut holes in the pipe to put the penetrameters into the pipe, following which fact holes had to be welded up. It is, however, a very serious problem. It actually did occur in a steam line that was going to operate in the neighborhood of 1800 psi. I am convinced that with a good welder you would be better off not to radiograph the weld, rather than to put 3-in. handholes in a pipe adjacent to the weld, which holes in turn have to be welded up. To put only the radium in would require a 1-in. hole; to get the penetrameters in would require a 2 to 3-in. hole. There is a big difference in the size of the hole and the size of the plug necessary, and the potential dangers.

Mr. O. R. Carpenter.[2]—For a good many years we have been building boilers with various types of headers and pipe, in which we customarily cut handholes over a large area in order to seal weld tubes. Some of these boilers work up to 2400-psi. pressure, and I do not believe we have ever had trouble with them. We customarily do the same thing in order to radiograph welds in pipe. We handle the hole merely by recessing the back and putting a cup-type handhole fitting in, which cannot therefore blow out. In fact, it only takes a seal weld to hold it; cutting a hole in pipe is quite feasible mechanically. The handhole has no need to be radiographed because of its size, and also because of the shoulder on it, which prevents any possibility of failure of the weld.

Mr. C. A. Adams.[3]—I should like to ask Mr. Doan whether there is any consistent relationship between the equivalent penetrameter and the normal one as between different thicknesses of wall or pipe. If there were a certain definite relationship, the equivalent penetrameter might be 40, 60, or 80 per cent for a given film distance.

Mr. Gilbert E. Doan.[4]—We have not plotted the results in that way, but with these observations of three different thicknesses it might be possible to find such a relationship. It would be worth trying.

Mr. H. H. Lester.[5]—A penetrameter placed on the wrong side will show visibility and contrast; it will show whether it is detectable or not, but it does not bring out what to my mind is one of the most essential features, which is the sharpness of outline. It is unfavorably placed to bring that out, which I think is one of the most essential points of the penetrameter, namely, the correctness of the d/t ratio used. Personally I would rather leave the penetrameter off altogether if it had to be placed on the wrong side.

Mr. J. P. Magos.[6]—Why not put a

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[1] Metallurgist, Pittsburgh Piping and Equipment Co., Pittsburgh, Pa.

[2] Engineer, The Babcock & Wilcox Co., Barberton, Ohio.

[3] Professor of Engineering, Emeritus, Harvard University, Cambridge, Mass.; and Consulting Engineer, Edward G. Budd Manufacturing Co., Philadelphia, Pa.

[4] Professor of Metallurgy, Lehigh University, Bethlehem, Pa.

[5] Principal Physicist, Watertown Arsenal, Watertown, Mass.

[6] Directing Engineer, Research Laboratories, Crane Co., Chicago, Ill.

penetrameter in a pipe line, tack-weld it in, then close the line with the circumferential weld and leave it there? Cut the small opening for introducing the radium, which according to Mr. Emerson is an easy matter, and leave the penetrameter in there. Worse things than that have been found in the pipe lines of power plants.

MR. CARPENTER.—That has been considered a number of times, and I think one of the primary objections is that the penetrameter will eventually corrode out and perhaps get into the turbine.

PRECISION RADIOGRAPHY—III

By Robert J. Schier[1] and Gilbert E. Doan[1]

This is the third of a series of papers on Precision Radiography, the first appearing in the ASTM Bulletin[2] and the second in the 1940 *Proceeding*.[3]

While a general equation has been developed for calculating radiographic distances,[4] this equation is rather cumbersome for ordinary radiographic problems and especially so for those in which the A.S.M.E. Code[5] is followed. A simpler equation can be developed for this case because, while the sizes and locations of flaws are variable, the diameters of the penetrameter holes are always the same fraction (4, 6, and 8 per cent) of the thicknesses of the test specimens and are always located on the source side of the specimen. These conditions simplify the equation substantially.

If, furthermore, a maximum radiographic angle can be adopted, such as for example the 2-deg. angle of Doan and Young,[4] and if the penetrameters lie on the shortest line between the source and the film, then the calculation of radiographic distances for A.S.M.E. exposures becomes quite simple and can be expresse by the equation:[6]

$$d = \frac{R + r}{\sin \alpha/2}$$

where:

d = the source-to-specimen distance;

R = the radius of the source;

r = the radius of the flaw, the smallest hole in a penetrameter that lies on the surface of the specimen nearest the source; and

α = the common vertex angle subtended by the source and the flaw.

Figure 1 shows diagrammatically a radiant source of radius R placed at a distance d from a specimen containing a flaw of radius r.

If, as is the case with A.S.M.E. penetrameters, the holes in the penetrameters increase in diameter as the specimen thickness increases and therefore the angular relationship is such that the registration is in major part by the umbral shadow, then the angular relationship between the source and the penetrameter holes (which determines the size of the penumbral shadow and hence the definition of the shadow image) will be nearly constant. In the general case, the flaws will not always fall on the shortest line between the source and the film. However, if the size of the flaws is comparable to that of the penetrameter holes, the flaws will be recorded with adequate definition because they are nearer the film and hence in a more favorable position for registry. Also, the angle between the flaws and the source will be smaller than the angle between the penetrameter holes and the source since the former are located at a greater distance from the source.

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[1] Gotshall Scholar in Metallurgy, and Professor of Metallurgy, respectively, Lehigh University, Bethlehem, Pa.

[2] Gilbert E. Doan, "Precision Radiography," ASTM Bulletin, No. 97, March, 1939, p. 27.

[3] Gilbert E. Doan and Winston H. Sharp, "Precision Radiography—II," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 1301 (1940).

[4] Gilbert E. Doan and Shang-Shoa Young, "Gamma-Ray Radiography," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part, II, p. 292 (1938).

[5] "Unfired Pressure Vessels," A.S.M.E. Code, p. 58 (1940).

[6] Proof of this equation will be found in the Appendix to this paper, see p. 1175.

To test this equation and to determine the angle α which should be adopted for certain registry of A.S.M.E. penetrameters lying on the shortest line between the source and the film a series of exposures was made using gamma rays from a spherical source having a working diameter of 3.0 mm.

EXPERIMENTAL METHODS

The source of gamma radiation was a spherical duralumin capsule containing 25 mg. of radium sulfate. The internal and external diameters of the capsule were 3.0 and 4.0 mm., respectively.

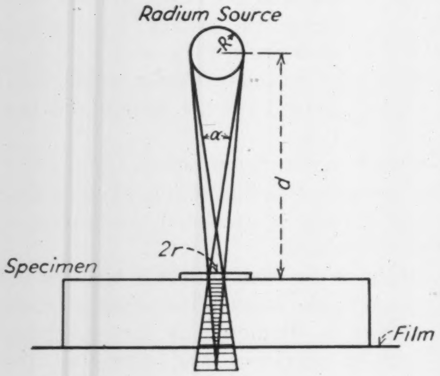

FIG. 1.

The test specimens were prepared by laying a weld bead with a 5 per cent chromium welding rod in a V-slot machined into the surface of $\frac{1}{4}$-in. steel plate, and different specimen thicknesses were obtained by backing up the face plates with mild steel blocks. A 5 per cent chromium welding rod was used to obtain a porous weld so that the equation developed could be tested with both natural flaws and A.S.M.E. penetrameters located adjacent to the weld bead. The weld metal was ground to remove irregularities on the weld surface so that it merged smoothly into the plate surface. A different face plate was used for each thickness of specimen, the specimen thicknesses being $\frac{1}{4}$, $\frac{1}{2}$, 1, $1\frac{1}{2}$, and 2 in.

TABLE I.—VERTEX ANGLE *VERSUS* VISIBILITY OF A.S.M.E. PENETRAMETERS.

Specimen thickness, ¼ in.

Film	Vertex Angle, deg.	Source-to-Film Distance, in.	Visibility Index of Penetrameter Holes[a]			Visibility Index of Weld Defects[a]
			1/16-in. Hole	1/16-in. Hole	1/16-in. Hole	
No. 251....	<¾	14	I	I	I	8
No. 252....	¾	11¾	I	I	I	8
No. 253....	1	8 3/16	I	I	I	7
No. 254....	1¼	6 9/16	I	I	I	7
No. 255....	1½	5½	I	I	I	7
No. 256....	1¾	4¾	I	I	I	7
No. 257....	2	4¼	I	I	I	6
No. 258[r]...	2¼	3¾				
No. 259....	2½	3 7/16	I	I	I	5
No. 2501...	3	2⅞	I	I	I	4
No. 2502...	3½	2½	I	I	I	3
No. 2503...	4	2¼	I	I	I	2
No. 2504...	4½	2	I	I	I	1
No. 2505...		11/16	I	I	I	1

[a] Numbers indicate relative visibility, 1—poor, 8—excellent, and I—invisible.

[r] Film ruined during development.

TABLE II.—VERTEX ANGLE *VERSUS* VISIBILITY OF A.S.M.E. PENETRAMETERS.

Specimen thickness, ½ in.

Film	Vertex Angle, deg.	Source-to-Film Distance, in.	Visibility Index of Penetrameter Holes[a]			Visibility Index of Weld Defects[a]
			1/16-in. Hole	1/16-in. Hole	1/16-in. Hole	
No. 50.....	½	16 3/16	I	I	I	8
No. 51.....		14	I	I	I	8
No. 52[r]....	¾	11⅛				
No. 53.....	1	8 7/16	I	I	I	8
No. 54.....		6½	I	I	I	7
No. 55.....	1½	5¾	I	I	I	7
No. 56[n]....		5 1/16				
No. 57.....		4¾	I	I	I	5
No. 58.....	2	4 7/16	I	I	I	5
No. 59.....	2¼	4	I	I	I	4
No. 501....	2½	3 11/16	I	I	I	3
No. 502...	3	3⅛	I	I	I	2
No. 503....	3½	2¾	I	I	I	2
No. 504...	4	2½	I	I	I	1
No. 505....		1⅜	I	I	I	1

[a] Numbers indicate relative visibility, 1—poor, 8—excellent, and I—invisible.

[n] No exposure made.

[r] Film ruined during development.

Source-to-film distances were calculated by means of the equation using vertex angles of from 4 to 1 deg. In every case the radius of the smallest hole in the penetrameter was used in the computations. Source-to-film distances are listed in Tables I to V.

TABLE III.—VERTEX ANGLE *VERSUS* VISIBILITY OF A.S.M.E. PENETRAMETERS.

Specimen thickness, 1 in.

Film	Vertex Angle, deg.	Source-to-Film Distance, in.	Visibility Index of Penetrameter Holes[a]			Visibility Index of Weld Defects[a]
			1/16-in. Hole	1/16-in. Hole	0.040-in. Hole[b]	
No. 10[n]....	½	19				
No. 11[n]....		14				
No. 12.....	¾	13 3/16	I	I	3	8
No. 13.....	1	10 1/8	I	I	3	8
No. 14.....		9 7/8	I	I	2	7
No. 15.....		8	I	I	2	7
No. 16.....	1½	7 1/16	I	I	2	7
No. 17[n]....		6 1/8				
No. 18.....	2	5 9/16	I	I	1	5
No. 19.....	2¼	5	I	I	I	4
No. 101....	2½	4 5/8	I	I	I	3
No. 102....	3	4	I	I	I	2
No. 103....	3½	3 9/16	I	I	I	1
No. 104....	4	3 1/4	I	I	I	1

[a] Numbers indicate relative visibility, 1—poor, 8—excellent, and I—invisible.
[b] Nearest drill sizes were used for holes.
[n] No exposure made.

TABLE IV.—VERTEX ANGLE *VERSUS* VISIBILITY OF A.S.M.E. PENETRAMETERS.

Specimen thickness, 1½ in.

Film	Vertex Angle, deg.	Source-to-Film Distance, in.	Visibility Index of Penetrameter Holes[a]			Visibility Index of Weld Defects[a]
			0.060-in. Hole[b]	0.090-in. Hole	0.120-in. Hole	
No. 150[n]...		21				
No. 151[n]...		15				
No. 152....	1	11 3/4	6	5	5	7
No. 153....	1½	8 5/16	5	5	5	7
No. 154[n]...	1¾	7 5/16				
No. 155....	2	6 5/8	5	4	4	6
No. 156....	2¼	6 1/16	3	3	3	6
No. 157....	2½	5 9/16	2	2	2	4
No. 158....	3	4 15/16	1	2	2	3
No. 159....	3½	4 7/16	1	1	1	2

[a] Numbers indicate relative visibility, 1—poor, 8—excellent, and I—invisible.
[b] Nearest drill sizes were used for holes.
[n] No exposure made.

The specimens were mounted on wooden stands and grouped around the source in the manner shown in Fig. 2. The face plates were oriented normal to the direction of propagation of the rays, and the surfaces containing the penetrameters and weld defects were oriented towards the source so that the flaws were in the most unfavorable position and so that the penetrameters fell on the shortest line between the source and the film.

TABLE V.—VERTEX ANGLE *VERSUS* VISIBILITY OF A.S.M.E. PENETRAMETERS.

Specimen thickness, 2 in.

Film	Vertex Angle, deg.	Source-to-Film Distance, in.	Visibility Index of Penetrameter Holes[a]			Visibility Index of Weld Defects[a]
			0.080-in. Hole[b]	0.120-in. Hole	0.160-in. Hole	
No. 20[n]....		21				
No. 21[n]....	1	13 7/16				
No. 22.....	1½	9 5/8	7	7	7	I
No. 23[n]....	1¾	8½				
No. 24.....	2	7 11/16	6	6	6	I
No. 25.....	2¼	7 1/16	5	5	5	I
No. 26.....	2½	6 9/16	3	3	3	I
No. 27.....	3	5 13/16	I	I	2	I
No 28.....	3½	5 1/4	I	I	I	I
No 29.....	4	4 7/8	I	I	I	I
No 201....		3 9/16	I	I	I	I

[a] Numbers indicate relative visibility, 1—poor, 8—excellent, and I—invisible.
[b] Nearest drill sizes were used for holes.
[n] No exposure made.

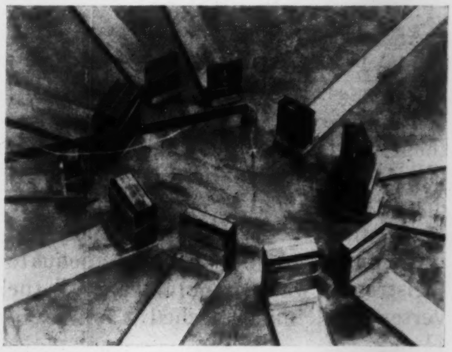

FIG. 2.—Experimental Arrangement.

The films were mounted in light-tight paper packs, each pack containing two films and three lead screens, and the packs were securely fastened to the backs of the specimens with rubber bands. A $\frac{1}{8}$-in. lead sheet served to give rigidity to the pack and to eliminate the effects of back scatter. Eastman's Ultra Speed Duplitized Film was used, and exposure times were calculated from charts given by Barrett, Gezelius, and Mehl.[7]

Duplicate films were viewed superimposed, and a visual comparison was made to determine what value of the vertex angle yielded a sharp image with a minimum exposure time.

RESULTS AND DISCUSSION

Tables I to V list the results obtained in this investigation. A visibility index has been devised and included in the tables to facilitate a comparison of the results. The visibility index indicates the relative ease with which the penetrameter holes were observed, and when reviewing the data it must be kept in mind that it was difficult to observe the sharpness of an image that was not readily detected in the radiograph.

An inspection of the data listed in Tables I to III reveals that for specimen thicknesses of $\frac{1}{4}$, $\frac{1}{2}$, and 1 in. the penetrameter holes could not be detected. For these thicknesses the comparison was based on the weld defects which had diameters of about $\frac{1}{16}$ in. or less but were deeper than the penetrameter holes. In the opinion of the authors adequate registration was not obtained until the vertex angle had reached a value of $1\frac{1}{2}$ deg.

An inspection of the data in Tables IV and V reveals that for specimen thicknesses of $1\frac{1}{2}$ and 2 in. adequate registration (visibility of 7) was not obtained until the vertex angle had reached a value of $1\frac{1}{2}$ deg. It is regretted that there was not sufficient time to make exposures at the 7 to 1 ratio prescribed by the A.S.M.E. code with low values of the vertex angle, as was done with the $\frac{1}{4}$, $\frac{1}{2}$, and 1-in. specimens, but with a vertex angle of $1\frac{1}{2}$ deg. all of the penetrameter holes were clearly outlined.

Recommended source-to-film distances, based on the results of this investigation for registration of A.S.M.E. penetrameters for a radium capsule the diameter of whose projected area is 3 mm., are listed in Table VI. If the assumption be made that the angular relationship determined holds true for specimen thicknesses greater than 2 in., then the radiographic distances for specimen thicknesses greater than 2 in. as shown in the table are obtained. It is expected that in the future this method will be extended to specimen thicknesses greater than 2 in. using capsules of different sizes.

TABLE VI.—SOURCE-TO-FILM DISTANCES RECOMMENDED FOR A.S.M.E. CONDITIONS FOR A CAPSULE OF RADIUM THE DIAMETER OF WHOSE PROJECTED AREA IS 3 MM.

Specimen Thickness, in.	Radiographic Distance, in. (= $1\frac{1}{2}$ deg.)
$\frac{1}{4}$	$5\frac{1}{2}$
$\frac{1}{2}$	$5\frac{3}{4}$
1	$7\frac{1}{16}$
$1\frac{1}{2}$	$8\frac{5}{16}$
2	$9\frac{5}{8}$
$2\frac{1}{2}$[a]	$10\frac{13}{16}$
3[a]	$12\frac{1}{8}$
$3\frac{1}{2}$[a]	$13\frac{3}{8}$
4[a]	$14\frac{5}{8}$

[a] For these thicknesses the assumption has been made that the angular relationship holds true for specimen thicknesses greater than 2 in.

An inspection of the radiographic technique normally used will reveal that the equation tested in this experiment is obvious. Nevertheless, it does appear that the equation is useful in permitting the reduction of exposure time to a minimum, when the A.S.M.E. Code is followed. The angular relationship required by the equation for specimen thicknesses up to 2 in. has been determined.

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[7] C. S Barrett, A. R. Gezelius, and R. F. Mehl, "The Technique of Radiography by Gamma Rays, "*Metals and Alloys*, Vol. 1, p. 872 (1930).

SUMMARY

1. The equation developed is a special application of the method used by Doan and Young[4] to A.S.M.E. conditions. It gives the minimum radiographic distance required for adequate definition of A.S.M.E. penetrameters when the penetrameters lie on the shortest line between the source and the film.

2. For the conditions examined, the results of this investigation show that adequate registration of A.S.M.E. penetrameters and weld defects is obtained with a vertex angle of $1\frac{1}{2}$ deg. for specimen thicknesses up to 2 in.

Acknowledgments:

The authors gratefully acknowledge the assistance of the Radium Chemical Co., the Bethlehem Steel Co., the Arcos Corp., and Joseph B. Reynolds, Professor of Mathematics, Lehigh University. The authors also are indebted for the fellowship under which this investigation was carried out provided by the endowment of Mr. William G. Gotshall.

APPENDIX

PROOF OF AUTHORS' EQUATION

From Fig. 3 it is readily seen that:

$$\sin \alpha/2 = \frac{R + r}{X}$$

and

$$d = X \cdot \cos \phi$$

$$d = \frac{R + r}{\sin \alpha/2} \cdot \cos \phi$$

When the penetrameter lies on the shortest line between the source and the film, the angle of incidence ϕ is zero, and the equation becomes:

$$d = \frac{R + r}{\sin \alpha/2}$$

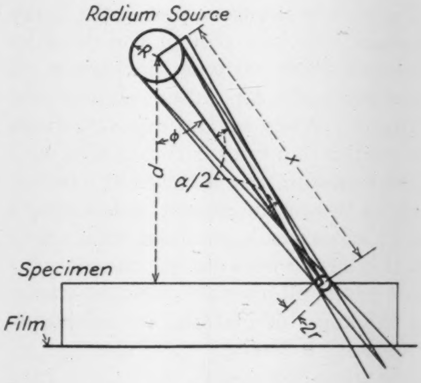

FIG. 3.

X-RAY FILM EVALUATION

By Vance Danford[1]

Synopsis

To use and improve the recording medium of industrial radiography more effectively, it is necessary to evaluate the characteristics of such material quantitatively.

This paper describes methods that have been worked out for interpreting the results of controlled X-ray exposures. The results are best shown and explained in graphical form.

Figure 1 is the basic plot of an X-ray exposure. It is a plot of the densities produced from various thicknesses of material with all other factors unchanged. When several exposure times are used, a family of curves is obtained. If the exposure time is varied as a factor, such as two or the square root of two, a family of curves is obtained with nearly equal intervals between the curves. Because of the filtering action of the changing thickness of material for nonmonochromatic radiation, this spacing interval increases slightly with increasing thickness of material.

A somewhat similar series of curves is obtained by varying the voltage and holding the time constant. In this case there is a more decided change in the shape of the curves due to the change in the quality of the radiation produced at the different voltages. This also is further modified by the changing thickness of material.

For a complete study of time and voltage changes, the desired range must be covered in intervals small enough for accurate interpretation. This plot is referred to as a density thickness plot.

Figure 2 gives a general picture of the over-all voltage-time variations. This technique curve is made from data obtained by a complete series of curves as described for Fig. 1. The voltage-time combinations are found for producing a given density at all the conditions covered.

From such a plot, various exposure combinations can be found which will produce the same density for the same thickness. For example, it is seen from Fig. 2 that a density of 1.00 can be produced for 0.5 in. of material at the following conditions:

Time, sec.	Primary voltage
120	56
60	63
30	70
15	82

Because it is possible to determine exposure conditions for a desired result, this type of plot is a means of presetting exposure techniques. This type of graph is satisfactory only if the material in question is of uniform thickness. It does not indicate the thickness that will be included in a desired density range.

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[1] Chief Research Engineer, Triplett & Barton Research Group, Triplett & Barton, Inc., Burbank, Calif.

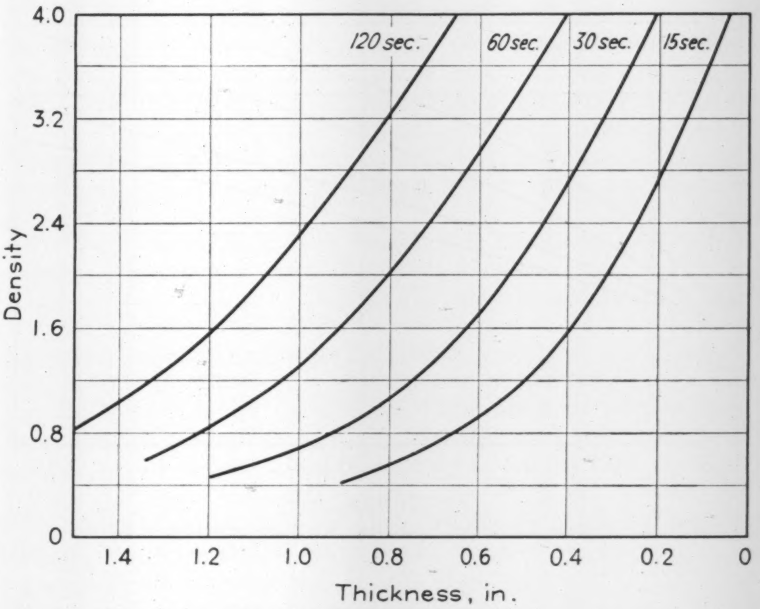

FIG. 1.—Basic X-ray Exposure.

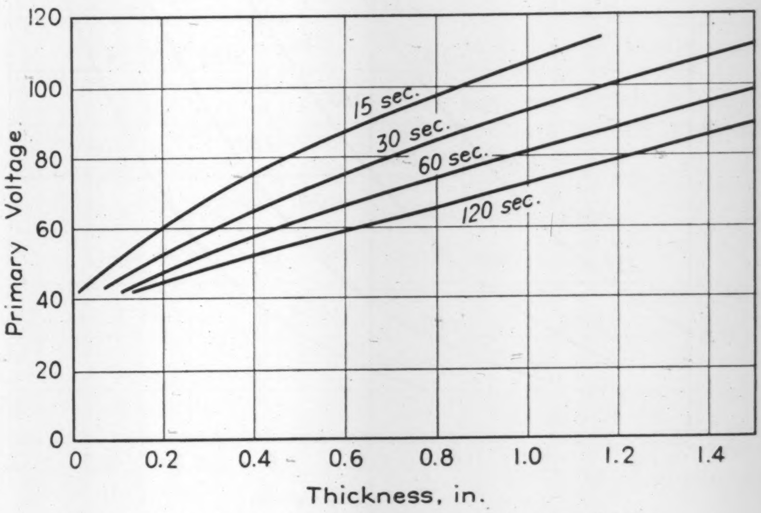

FIG. 2.—Voltage-Thickness Relation. Density = 1.00.

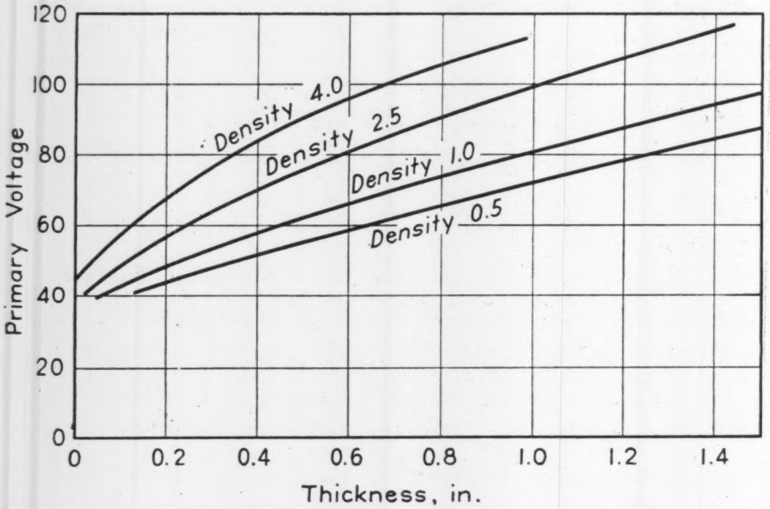

FIG. 3.—Voltage-Thickness Relation. Constant exposure = 600 mas.

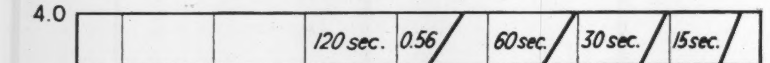

FIG. 4.—Basic X-ray Exposure, with Contrast Values.

Figure 3 is a technique curve similar to Fig. 2. The 1.0 density line is the same in this figure as the 60-sec. line in Fig. 2. The data for this line is obtained in the same manner. Instead of plotting lines for different times of exposure, they are plotted for the same time of exposure and different densities. These densities are usually the limiting densities due to the film contrast and reading equipment and also the most desirable average density.

This plot can be used very satisfactorily for setting exposure techniques of parts with varying thicknesses, as it indicates the thickness that can be included between the density limits chosen. For example, if a part varied between 0.4 in. and 0.8 in., it is seen that for 0.4 in. of material a primary voltage of 84 is necessary to produce a density of 4.0. A density of 4.0 is the upper density limit in this case. It is also seen that a voltage of 74 is needed to produce a density of 1.0 for 0.8 in. The density 1.0 is the lower density limit in this case. The voltage can be varied between these two limits. The voltage that would be chosen would be that which puts the largest or most important area at 2.5 which is the most advantageous density. This is the type of plot we most commonly use for setting exposure techniques.

This plot, as shown in Fig. 3, does not indicate the sensitivity of the resulting picture. To select the most desirable density and the lower density limit, the sensitivity or radiographic contrast obtained must be determined.

Figure 4 is the same as Fig. 1 except that it has digits along the curves expressing the radiographic contrast at those points. The quantitative expression of radiographic contrast is the percentage of the total thickness that will produce a just visible density difference of 0.02. This percentage represents the percentage of the total thickness that would be just detectable in the interpretation of a radiograph. There is a minimum density difference the eye can detect. Under favorable laboratory conditions this minimum value is about 0.005. For calculating radiographic contrast applicable to production conditions, the conservative density difference of 0.02 is used.

The value is calculated by assuming a straight-line relationship between two densities, usually those produced by a thickness difference of 0.10 in. of material. The thickness represented by a density difference of 0.02 is calculated and divided by the total average thickness over which the density difference was measured. This is multiplied by 100 to express it in per cent.

An example of a radiographic contrast calculation is the following: if it is found that 0.10 in. of material will produce a density difference of 0.20, then a density difference of 0.02 will represent 0.01 in. of material. If the average total thickness over which the density was measured was 1.00 in., then 0.01 in. would be 1.0 per cent of the total thickness. This percentage is used to express the radiographic contrast.

This radiographic contrast calculation formula is as follows:

Radiographic contrast

$$= \frac{\Delta T \times 0.02 \times 100}{\Delta D \times T}$$

where:

ΔT = included thickness over which the density was measured,

T = average total thickness over which the density was measured,

ΔD = density difference over a known thickness change, and

0.02 = minimum density difference assumed to be detectable.

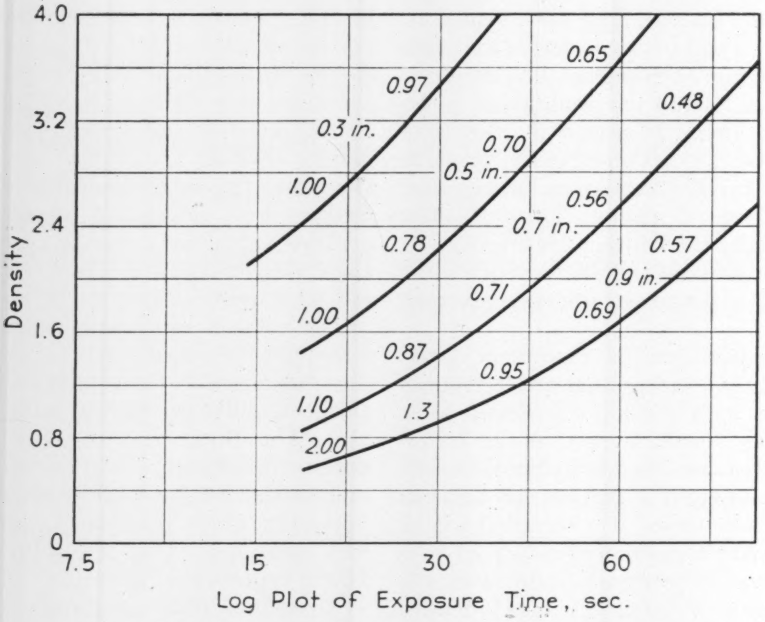

FIG. 5.—Density *versus* Exposure Time, for Constant Thickness.

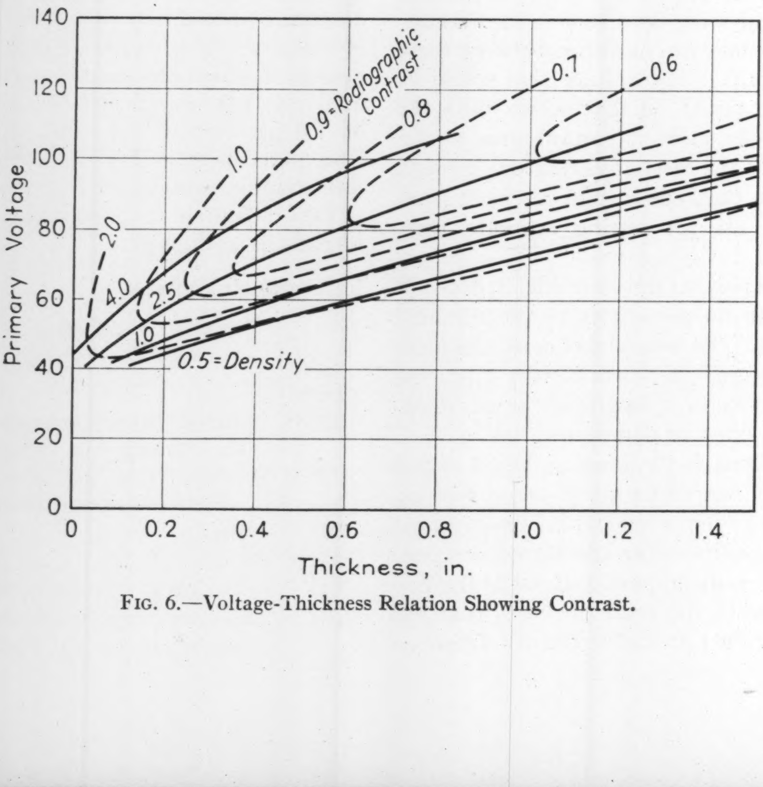

FIG. 6.—Voltage-Thickness Relation Showing Contrast.

From plots similar to Fig. 4, radiographic contrast can be replotted on the appropriate technique curve.

Figure 5, the characteristic curve of a radiographic film, shows the variation of radiographic contrast with density. Each curve represents constant thickness. This plot is made from Fig. 4 using the density-time relations found for a constant thickness.

It is seen that radiographic contrast improves with greater density for constant voltage. This continues to a point where it is no longer readable due to the light source.

From a plot of this nature, the exact exposure required for any given density for a given thickness can be determined. Such a value is used to represent the film speed. For our purposes we express the film speed as the mas. necessary to produce a density of 2.5 for 0.5 in. of a specified material and at a given voltage or quality of radiation.

Figure 6 is the same as Fig. 3 except dotted lines representing the trend of the radiographic contrast are added. From the figure it is seen that at constant time the best radiographic contrast is reached at a density of 2.5. It also shows that above a density of 1.0, for the particular film used, the radiographic contrast is generally better than 1 per cent.

The statement that the best density is 2.5 for constant exposure is not to be confused with the statement that the radiographic contrast continues to improve with increasing density as given for Fig. 5. The latter statement applies to constant voltage, and increasing the density by exposure time rather than by voltage.

Another effect to be considered relating to density and radiographic contrast is that the high densities represent the thinner sections of the part. The density thickness curve is steeper at the high

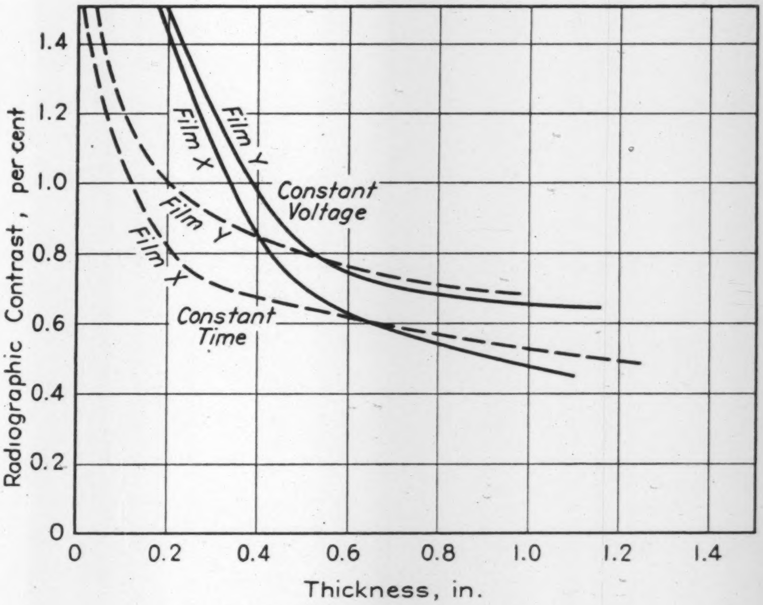

FIG. 7.—Radiographic Contrast Comparison.

densities which means smaller thickness differences can be detected. When these thickness differences are expressed as a percentage of the total thickness where they occur, the percentage is usually greater at these thinner sections, which means less radiographic contrast. From a curve of this nature, technique can be set so that the density and thickness limits can be considered and the resulting radiographic contrast within these limits will be known.

Figure 7 is an evaluation of radiographic contrast. It is obtained from data similar to Fig. 4, plotting the radiographic contrast obtained for a given density at a given voltage. This gives radiographic contrast comparison at constant voltage. A plot similar to Fig. 4, with time constant and voltage varied, will give radiographic contrast comparison at constant time as shown in Fig. 7.

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